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TRANSACTIONS

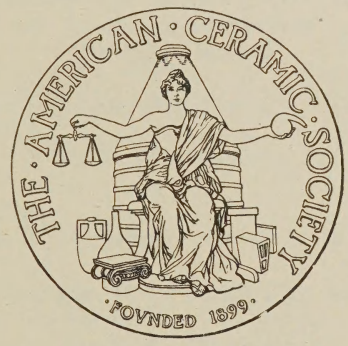
OF THE

American Ceramic Society

VOLUME XVIII

Containing the Papers Read at the Meeting Held at Cleveland, Ohio, February 21, 22, 23, and 24, 1916, Together with Supplementary Discussions and Other Matters.

ARTHUR S. WATTS, *Editor*



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RAINEY, L. B., CR.E., 42 Sherman Ave., Mansfield, Ohio	
RAMSAY, ANDREW, Mt. Savage, Md.	The Andrew Ramsay Company
RAMSAY, JOHN D., St. Marys, Pa.	President and General Manager, Elk Fire Brick Company
RANCKE, LOUIS N., 45 Knickerbocker Bldg., Baltimore, Md.	
RANDALL, JAMES E., Indianapolis, Ind.	Junior Editor, *Clayworker*
*RANDALL, THEODORE A., Indianapolis, Ind.	*Editor, Clayworker*
RATHFON, WILLIAM OWEN, B.S., 203 N. Chicago Ave., Brazil, Ind.	Chicago Fire Brick Company
RATHJENS, GEORGE WILLIAM, B.S., C.E., St. Paul, Minn.	Superintendent, Twin City Brick Company
RAYMOND, GEORGE M., 1511 Grand Ave., Dayton, Ohio	General Manager, C. W. Raymond Company
REA, WILLIAM J., Buffalo, N. Y.	Superintendent, Buffalo Pottery Company
REED, CHAS. S., 832 W. Adams St., Chicago, Ill.	President Chicago Retort and Fire Brick Company
REEVE, HOWARD T., F.I.C., South Bridge, Mass.	Chief Scientist, American Optical Company
REYNOLDS, PIERCE B., A.B., 433 Wyoming Ave., Kingston, Pa.	Treasurer, Dorrance Terra Cotta Company
RHEAD, FREDERICK HURTEN, Santa Barbara, Calif.	Editor and Manager, Potter Publishing Company
RHOADES, RALPH STANLEY, St. Louis, Mo.	General Manager, Evens and Howard Fire Brick Company
RICHARDSON, ERNEST, Beaver Falls, Pa.	Vice-President and Treasurer, Ingram-Richardson Manufacturing Company
RIESS, DEWITT F., Sheboygan, Wis.	Secretary and Factory Manager, Vollrath Company
RIVERS, WILLIAM EDWARD, Old Bridge, N. J.	Vice-President, Old Bridge Enameled Brick and Tile Company

* Life member

ROBERTSON, FRED. H., 809 N. Alvarado St., Los Angeles, Cal.	Los Angeles Press Brick Company
ROBERTSON, HUGH SCHUYLER, B.S., 211 Sycamore St., Pittsburgh, Pa.	Harbison-Walker Refractories Company
ROBINEAU, S., 108 Pearl St., Syracuse, N. Y.	President, Keramic Studio Publishing Company
ROBINSON, LOUIS G., M.E., 4th and Park Aves., Newport, Ky.	
ROCKHOLD, KENNETH EDWARD, B.S., Zanesville, Ohio	L. E. Rodgers Engineering Company
RODGERS, EBEN, Alton, Ill.	Secretary and Treasurer, Alton Brick Company
ROESSLER, FRANZ, 39 High St., Perth Amboy, N. J.	Roessler and Hasslacher Chemical Company
ROGERS, GREGORY L., Danville, Ill.	American Refractories Company
ROSE, ARTHUR VEAL, 11 E. Thirty-sixth St., New York, N. Y.	General Manager, Haviland and Company
RUSOFF, SAMUEL, Anderson, Ind.	National Tile Company
RYAN, JOHN J., B.S., Seguin, Texas	Secretary, Seguin Brick and Tile Company
SALISBURY, BERT E., 1810 W. Genesee St., Syracuse, N. Y.	President, Onondaga Pottery Company
SANDERS, JOHN W., Moundsville, W. Va.	Enameler, United States Stamping Company
SANT, THOMAS HERBERT, East Liverpool, Ohio	Secretary-Treasurer, The John Sant and Sons Company
SAUNDERS, WILLIAM EDWARD, B.S., E.M., 1401 Arch St., Philadelphia, Pa.	Engineer, United Gas Improvement Company
SCHACHTEL, S. H., CR.E., Coffeyville, Kansas	Ludowici-Celadon Company
SCHAPPER, WALTER, 192 N. Clark St., Chicago, Ill.	Salesman, Roessler and Hasslacher Chemical Company
SCHAULIN, GEORGE MARVIN, Mazon, Ill.	
SCHURECHT, HARRY GEORGE, Lord Hall, O. S. U., Columbus, Ohio.	United States Bureau of Mines
SEAVER, KENNETH, S.B., Pittsburgh, Pa.	Assistant General Sales Manager, Harbison-Walker Refractories Company
SEBRING, CHARLES L., Sebring, Ohio	General Manager, Sebring Pottery Company
SHEERER, MARY G., Newcomb Pottery Company, New Orleans, La.	Assistant Director, Newcomb Pottery
SHOEMAKER, GEORGE W., Room 305, 20 E. Broad St., Columbus, Ohio	

SINCLAIR, GEORGE E., Trenton, N. J.	Secretary, Star Porcelain Company
SINCLAIR, HERBERT, Trenton, N. J.	General Manager, Star Porcelain Company
SINGER, L. P., Lincoln, Calif.	Chemist, Gladding, McBean and Company
SMITH, AMEDEE M., Portland, Oregon	President Western Clay Manufacturing Company
SMITH, HARRY W., 314 Smith St., East Liverpool, Ohio	Representative, Roessler and Hasslacher Chemical Company
SMITH, JAMES M., New Castle, Pa.	Treasurer, Shenango Pottery Company
SMITH, NORMAN G., Brunswick, Me.	Treasurer and General Manager, Maine Feldspar Company
SMITH, PERRY A., New Brighton, Pa.	Secretary, A. F. Smith Company
SOLON, ALBERT L., Fairfax, Calif.	Manager, Arequipa Pottery
SOLON, MARC, Trenton, N. J.	General Manager and Secretary Mercer Pottery Company
SPEIR, HARRY F., Cedarville, N. J.	President, New Jersey Pulverizing Company
SPIKER, JOHN A., 811 Kearns Bldg., Salt Lake City, Utah	General Manager, United States Fire Clay Company
SPRINGE, OTTO, B.S., Galesburg, Ill.	Ceramic Engineer, Purington Paving Brick Company
SPROAT, IRA E., Dry Branch, Ga.	
SPURRIER, HARRY, 704 Sheridan Ave., Detroit, Mich.	Chemist, Jeffrey-Dewitt Company
STAMM, JOHN, 859 Ohio Ave., E. Liverpool, Ohio	Superintendent, The Croxall Chemical and Supply Company
STANGER, FREDERICH, 520 Walnut St., Philadelphia, Pa.	Sales Manager, Enterprise White Clay Company
STANGL, MARTIN, Dundee, Ill.	Haeger Brick and Tile Company
STANLEY, WILLIAM W., PH.B., Great Barrington, Mass.	Manager, Stanley Insulating Company
STAPLER, WALTER STEVENS, Stevens Pottery, Ga.	Assistant General Manager, Stevens Brothers and Company
STAUDT, AUGUST, Perth Amboy, N. J.	President, Perth Amboy Tile Works
STEINMAYER, R. A. J., B.S., LaSalle, Ill.	LaSalle Pressed Brick Company
STERN, NEWTON W., 67 New Montgomery St., San Francisco, Calif.	Secretary and Treasurer, Pacific Porcelain Ware Company
STEVENS, DOUGLAS F., M.E., Cayuga, Indiana	Superintendent, Acme Brick Company

Stevenson, Wm. G., Oregon, Ill.	Secretary-Treasurer, Ohio Silica Company
Stowe, Charles Brown, Cleveland, Ohio	President, National Fire Brick Company
Swalm, Phaon H., Trenton, N. J.	Assistant Superintendent, Trenton Fire Clay and Porcelain Company
Tamlyn, Walter Irving, E.E., 359 Crown St., Brooklyn, N. Y.	Assistant Engineer, with Ralph D. Mershon.
Tatton, Joseph, 206 Academy St., Trenton, N. J.	Trenton Potteries Company
Taylor, Royal W., 707 Twelfth St., N. W., Canton, Ohio	Assistant Secretary and Chemist, The Canton Stamping and Enameling Company
Tefft, Charles Forest, B.S. (in Cer.), Darlington, Beaver Co., Pa.	Manager, Darlington Clay Products Company
Thomas, Chauncey Rapelje, 2336 San Pablo Ave., Berkeley, Calif.	
Thomas, David C., Wellsville, Ohio	Ceramic Chemist, U. S. Pottery Company
Thomas, George E., St. Louis, Mo.	General Superintendent, Highlands Fire Clay Company
Thomas, James Ray, B.S.(in E.E.), Crawfordsville, Ind.	Manager, Standard Brick Company
Thrall, Charles U., 1170 Broadway, New York, N. Y.	Superintendent, Atlantic Terra Cotta Company
Tillotson, Edwin W., Jr., B.A., Ph.D. Pittsburgh, Pa.	University of Pittsburgh
Tilton, Earl Monroe, Cr.E., 96 W. 9th Ave., Columbus, Ohio	Assistant Testing Engineer, State Highway Department
Timmerman, Walter F., Kansas City, Kansas	Superintendent, Western Terra Cotta Company
Tone, Frank J., Niagara Falls, N. Y.	Works, Manager, Carborundum Company
Townsend, Everett, 344 Bellevue Ave., Trenton, N. J.	General Manager, Robertson Art Tile Company
Trautwein, J. O., 417 S. Dearborn St., Chicago, Ill.	President, Trautwein Dryer and Engineering Company
Treischel, Chester, B.S., 127 S. Chicago Ave., Kankakee, Ill.	Kankakee Brick and Tile Company
Truman, Gail R., 5801 Manchester Ave., St. Louis, Mo.	Ceramic Chemist, St. Louis Terra Cotta Company
Turner, A. M., M.E., Sciotoville, Ohio	Superintendent and Treasurer, Scioto Fire Brick Company
Van Schoik, Elmer Holmes, B.S., (in Cer.), Box 166, Ottawa, Ill.	Chicago Retort and Fire Brick Company
Vodrey, William E., East Liverpool, Ohio	General Manager, Vodrey Pottery Company

VOLLKOMMER, JOSEF, Bessemer Bldg., Pittsburgh, Pa.	President and Manager, The Vitro Manufacturing Company
VOLZ, WILLIAM J., St. Louis, Mo.	Superintendent, Evens and Howard Fire Brick Company
WAGNER, FRITZ, 1000 Clybourn Ave., Chicago, Ill.	Vice-President and Treasurer, The Northwestern Terra Cotta Company
WALKER, CHARLES HENRY, 400 W. 4th St., East Liverpool, Ohio	Assistant Superintendent, Homer Laughlin China Company
WALKER, F. WM., JR., B.S. (in M.E.), Beaver Falls, Pa.	Superintendent, Beaver Falls Art Tile Company
WANKA, F. O., Clare and Wiscomico Sts., Baltimore, Md.	Maryland Terra Cotta Company
WARD, J. W., 628 E. Main St., Latrobe, Pa.	General Superintendent, Pittsburgh High Voltage Insulator Company
WEIGEL, CHARLES, Hebron, North Dakota	President, Hebron Fire and Pressed Brick Company
WEIL, EDGAR, H., B.S., Cleveland, Ohio	Secretary and Works - Manager, Enamel Products Company
WELLS, HARRY B., Canton, Ohio	Superintendent, Belden Brick Company
WHITAKER, FRED. A., Keasbey, N. J.	Superintendent, German - American Stoneware Works, Plant No. 1
WHITE, RAY HILL, A.B., S.B., Niagara Falls, N. Y.	Research Engineer, Norton Company
WHITELAW, JAMES CAMERON, B.S., 1220 Boren Ave., Seattle, Wash.	
WHITFORD, WILLIAM GARRISON, PH.B. (in Cer.), Chicago, Ill.	Instructor in Design and Ceramic Art, University of Chicago
WHITMER, J. D., CR.E., 9 Ball St., Zanesville, Ohio	American Encaustic Tile Company
WIGFIELD, CLAUDE LAWRENCE, 205 E. 2nd St., Elyria, Ohio	Enameler, Enamel Pipe and Engineering Company
WILDER, THOMAS MILTON, Ravinia, Ill.	Proprietor, Wildwood Shop
WILL, OTTO W., Perth Amboy, N. J.	Superintendent, Color Department, Roessler and Hasslacher Chemical Company
WILSON, HEWITT, CR.E., Auburn, Washington	Ceramic Chemist, Northern Clay Company
WOODS, WILLIAM J., A.B., Lewistown, Pa.	Assistant General Manager, Pennsylvania Pulverizing Company
WORTH, S. HARRY, 706 Franklin Bank Bldg., Philadelphia, Pa.	President, Pennsylvania Feldspar Company
WRIGHT, JOSEPH WILLIAM, 511 E. Green St., Champaign, Ill.	Student, Ceramic Engineering, University of Illinois

YATES, ALFRED,
Shawmut, Elk County, Pa.
General Manager, Shawmut Paving Brick Company

YEARSLEY, HOWARD L.,
133 Wesmont Ave., Haddonfield, N. J.
Manager, Electrical Porcelain and Manufacturing Company

YOUNG, GEORGE F.,
Zanesville, Ohio
Manager, Roseville Pottery Company

YOWELL, JOHN BENNETT, B.S.,
Dudley, Ill.

ZOPFI, ALBERT S.,
Toledo, Ohio
Secretary and Treasurer, Buckeye Clay Pot Company

RULES OF THE SOCIETY

[Revised 1915]

I

OBJECTS

The objects of the American Ceramic Society are to promote the arts and sciences connected with ceramics by means of meetings for the reading and discussion of professional papers, the publication of professional literature, and for social intercourse.

II

MEMBERSHIP

The Society shall consist of Honorary Members, Active Members, Associate Members and Contributing Members.

Honorary members must be persons of acknowledged professional eminence, whom the Society wishes to honor in recognition of their achievements in ceramic science or art. Their number shall at no time exceed two percent of the combined active and associate membership.

Active members must be persons competent to fill responsible positions in ceramics. Only Associate Members shall be eligible to election as Active Members, and such election shall occur only in recognition of attainments in the field of ceramics, and interest in the Society, as evidenced by papers or discussions contributed to its meetings.

Associate Members must be persons interested in ceramics or allied arts.

Contributing Members must be persons, firms, or corporations who, being interested in the Society, make such financial contributions for its support as are prescribed in Section 3.

Honorary Members shall be nominated for election by at least five active members, and approved by the Board of Trustees. Their nomination shall be placed before the Society at an annual meeting, and to be elected they must receive the affirmative vote of at least 90 percent of those voting, by letter ballot, at the next succeeding annual meeting.

To be promoted to active membership, Associate Members must be nominated in writing by an Active Member, and must be seconded

by not less than two other Active Members, and must be approved by the Board of Trustees. Their nomination shall be accompanied by a statement of their professional qualifications and a list of their publications, if any. Their nominations, when approved by the Board of Trustees, shall be placed before the Society at an annual meeting, and to be elected they must receive the affirmative vote of not less than 75 percent of those voting, by letter ballot, at the next succeeding annual election.

A candidate for Associate Membership must make application upon a form, prepared by the Board of Trustees, which shall contain a written statement of the age and professional experience of the candidate, and a pledge to conform to the laws, rules and requirements of the Society. Such applications must be endorsed by two Active Members of the Society as sponsors, and must be approved by the Board of Trustees. The Board may act by letter ballot upon such application at any time, after which an approved candidate may be enrolled on the proper list of the Society upon payment of the fees and dues prescribed in Section 3.

Contributing Members shall be nominated by an Active Member, and approved by the Board of Trustees, and may be enrolled on the proper list of the Society at any time upon payment of the dues prescribed in Section 3.

All Honorary Members, Active Members, Associate Members, and Contributing Members shall be equally entitled to the privileges of membership, except that only Active Members shall be entitled to vote and hold office. The roster of each grade of membership shall be printed separately, in at least one publication issued by the Society annually. Any person may be expelled from any grade of the membership of the Society if charges signed by five or more active members are filed against him, and a majority of the Board of Trustees examines into said charges and sustains them. Such person, however, shall first be notified of the charges against him, and be given a reasonable time to appear before the Board of Trustees, or present a written defense, before final action is taken by the Board of Trustees.

III

DUES

Honorary Members shall be exempt from all fees or dues.

The initiation fee of Active Members shall be ten dollars, and if not paid within three months after the date of their election, the latter shall be null and void. The annual dues for Active Members shall be fixed by the Board of Trustees, and shall not exceed ten dollars.

The initiation fee of Associate Members shall be five dollars, and their annual dues shall be fixed by the Board of Trustees, but shall not exceed five dollars. The privileges of Associate Membership after

election shall begin upon payment of the initiation fees, and dues for the first year.

Contributing Members shall pay no initiation fee into the Society. Their annual dues shall be fixed by the Board of Trustees, but shall not exceed twenty-five dollars. The privileges of membership shall begin upon payment of the annual dues.

Any Active Member or Associate Member in arrears for over one year may be suspended from membership by the Board of Trustees, until such arrears are paid, and in event of continued dereliction, may be dropped from the rolls. Active Members in arrears are not eligible to vote. The annual dues of Active and Associate Members are payable within three months succeeding date of annual meeting.

IV

OFFICERS

The affairs of the Society shall be managed by a Board of Trustees, consisting of a President, Vice-President, Secretary, Treasurer and three Trustees, who shall be elected from the Active Members at the annual meeting, and hold office until their successors are elected and installed.

The President, Vice-President, Secretary and Treasurer shall be elected for one year, and the Trustees for three years; and no President, Vice-President, or Trustee shall be eligible for immediate re-election to the same office.

The duties of all officers shall be such as usually pertain to their offices, or may be delegated to them by the Board of Trustees or the Society; and the Board of Trustees may, at its discretion, require bonds to be furnished by the Treasurer.

Vacancies in any office shall be filled by appointment by the Board of Trustees, but the new incumbent shall not thereby be rendered ineligible to re-election at the next annual meeting to the same office. On the failure of any officer to execute his duties within a reasonable time, the Board of Trustees, after duly warning such officer, may declare the office vacant, and appoint a new incumbent.

A majority of the Board of Trustees shall constitute a quorum, but the Board of Trustees shall be permitted to carry on such business as it may desire by letter.

V

ELECTIONS

At the annual meeting, a Nominating Committee of five Active Members, not officers of the Society, shall be appointed, and this committee shall send the names of the nominees to the Secretary at least sixty days before the annual meeting who shall immediately forward the same to the Active Members. Any other five Active

Members may act as a self-constituted Nominating Committee, and also present the names of any nominees to the Secretary, provided it is done at least thirty days before the annual meeting. The names of all nominees provided their assent has been obtained, shall be placed on the ballot without distinction as to nomination by the regular or self-constituted Nominating Committee, and shall be mailed to every member, not in arrears, at least twenty days before the annual meeting. The ballots shall be enclosed in an inner blank envelope, and the outer envelope shall be endorsed by the voter, and mailed to the Secretary. The blank envelopes shall be opened by three scrutineers appointed by the President, who will report the result of the election at the last session of the annual meeting. A plurality of votes cast shall elect.

VI

MEETINGS

The annual meeting shall take place on the first Monday in February, or as soon thereafter as can be conveniently arranged, at such place as the Board of Trustees may decide, at which time a report shall be made by the Board of Trustees, Treasurer and scrutineers of election, and the accounts of the Treasurer shall be audited by a committee of three, appointed by the President. Ten Active Members shall constitute a quorum at any regular meeting, and a majority shall rule unless otherwise specified.

The order of business at the annual meeting shall be:

1. President's address.
2. Reading of minutes of last meeting.
3. Reports of the Board of Trustees and Treasurer.
4. Appointment of committees.
5. Old business.
6. New business.
7. Reading of papers.
8. Announcement of election of officers, Honorary and Active Members.
9. Installation of officers and new members.
10. Adjournment.

Other meetings may be held at such time and places during the year as the Board of Trustees may decide, but at least twenty days' notice shall be given of any meeting.

The President shall appoint at the annual meeting a committee of five, to be known as the Summer Meeting Committee, whose duty it shall be to arrange for a summer excursion meeting at some suitable point. The expenses of the Summer Meeting Committee in arranging the program of visits and for printing, rooms and facilities for meetings, shall be borne by the Society.

VII

PUBLICATIONS

The Board of Trustees shall employ, at suitable compensation, an Active Member of the Society as Editor of its publications. The Editor, together with the Secretary and Treasurer, shall constitute a Publication Committee. The Publication Committee shall provide for the publication of an annual volume, entitled "Transactions of the American Ceramic Society." This volume shall contain a list of the officers, a list of members of the Society, classified into grades, a list of the ex-Presidents, the dates and location of meetings, the annual report of the Board of Trustees and Treasurer, the list of prices of the publications of the Society, the rules of the Society, and any other matter pertaining to the business administration of the Society that the Publication Committee may think proper. It shall also contain such of the papers and discussions thereon as the Publication Committee may consider desirable, and each volume shall contain a list of the papers and discussions and an index. The volume shall be six inches wide by nine inches long, and part of the issue shall be bound in paper covers and part in cloth binding. The quality of the paper, the kind of type, the illustrations, and all other mechanical details of the printing and publishing of the books or reprints shall be in the hands of the Publication Committee, subject to the control of the Board of Trustees.

The acts and policies of the Publication Committee shall at all times be subject to the examination and approval of the Board of Trustees, but the Board shall be bound by contracts entered into by the Publication Committee in the name of the Society. The Publication Committee shall have full power and authority to decide what papers and discussions to publish, which discussions shall be germane to the subject matter, and in what order they shall be published, and in what manner and to what extent they shall be illustrated. In event that the Board of Trustees shall undertake the publication of some other matter or book than the Transactions, the Publication Committee shall act in the same capacity for that publication as in the publication of the annual volume of the Transactions.

One copy of the Transactions shall be sent prepaid to each member of the Society not in arrears. No member shall be furnished more than one copy of the Transaction free for any single year. Members cannot purchase extra copies of the Transaction at less than the current commercial rates. A member shall be permitted to purchase one complete file of the publications of the Society at less than the current commercial rate, the amount to be fixed by the Publication Committee and called the Member's rate.

The Secretary shall have the custody of all publications of the Society, shall keep them safely stored and insured, and shall sell these

volumes to the public at prices which shall be fixed by the Publication Committee for each new volume as issued. The Publication Committee shall also, from time to time, fix the price of the old volumes remaining unsold, and shall have authority to refuse to sell the old volumes of the Transactions except in sets, at such time as the quantity remaining of any number becomes so small, as in their judgment to warrant such action.

The Editor shall request the author of each article appearing in the Transactions of the Society to fill out and sign, with a definite time limit, a blank form, specifying the number of reprints of said article, if any, which he desires. This form shall contain a table from which can be computed the approximate cost at which any reprints will be furnished. In event that the expense of furnishing the desired number of reprints is large, the Publication Committee may require the author to pay in advance for part or all of the cost involved before the publication of reprints is begun. On receipt of such signed order within the time limit set, the Editor shall cause to have printed the desired number of copies. If the author makes no reply, or replies after the time limit has expired, then the Society will not be responsible for the publication of any reprints of the article in question, except at the usual market price for the printing of new matter.

No one shall have the right to demand the publication of an article independent of the discussion which accompanied it, and no one having taken part in a discussion upon an article shall be entitled to order reprints of the discussion separately and apart from the article itself.

The Society is not, as a body, responsible for the statements of facts or opinions expressed by individuals in its publications.

VIII

PARLIAMENTARY STANDARD

Roberts "Rules of Order" shall be the parliamentary standard on all points not covered by these rules.

IX

AMENDMENTS

To amend these rules, the amendment must be presented, in writing, at the annual meeting of the Society, and must be printed on the ballot for officers and sent out not less than twenty days in advance of the next annual meeting, and if the said letter ballot shows an affirmative vote of not less than two-thirds of the total vote cast, then the same shall be declared carried.

X

LOCAL SECTIONS

Designation. Local Sections, each carrying some distinguishing title prefixed to the words "Section of the American Ceramic Society," may be authorized by the Society.

Purpose. The purposes of such sections shall be to strengthen and extend the work of the Society as defined in Section I of its Rules, by more frequent meetings in local centers than are possible to the Society as a whole, and by bringing the benefits of the work to persons who would not otherwise be reached.

Granting of Charters. Application for permission to form a local section must be in writing and signed by not less than ten members of the Society in good standing, residing in the general locality where the section is to be formed, of whom one at least shall be an Active member. To be considered at any given meeting, an application must be filed with the secretary at least 30 days prior to the date of the meeting, and notice that the application is pending must appear in the program of the meeting. To be granted, the application must receive the affirmative vote of two-thirds of those present. In event of affirmative action, the Society will issue a charter to the applicants, authorizing them to form a section under the name proposed. Charters for local sections may be temporarily suspended by the Board of Trustees for cause, but no charter can be permanently rescinded, except by vote of two-thirds of those present at a regular meeting of the Society after due publication in the program of the meeting that the matter is pending.

Legislative Powers. Local sections shall have power to make their own rules and by-laws, except that they shall not pass any rule or by-law which is in conflict with the Rules of the Society.

Officers. The Officers of local sections shall be a Chairman, a Secretary and a Councilor, and such others as the Section may prescribe. The duties of the Chairman and Secretary shall be such as usually pertain to those offices. The Councilor must be an Active member of the Society, and it shall be his duty to advise the Section in all matters pertaining to its relations with the Society, and to make an annual report to the Society regarding the work and status of the Section. The names of the Chairman, Secretary and Councilor of each Section shall appear in the roster of the Society in the Annual Volume of the Transactions of the Society.

Eligibility. Only members of the Society shall be eligible to full participation in the work of local Sections, but Sections may provide in their by-laws for a limited participation in their work by persons not members of the Society, when in their opinion the purpose of

the Society will be subserved by such a course, provided always that such persons shall not vote or hold office in the Section.

Finances. Local Sections shall have power to fix their own dues or assessments, such dues or assessments being in addition to and independent of the regular dues and assessments of the Society upon its members. No Section shall have authority to incur debt in the name of the Society or for which the Society may become liable.

XI

STUDENT BRANCHES

Designation. Student Branches, each carrying some distinguishing title prefixed to the words "Student Branch of the American Ceramic Society," may be established in Institutions in which regular courses of instruction in Ceramics are maintained.

Purpose. The purposes of such Student Branches shall be to strengthen and extend the work of the Society, as defined in Section I of the Rules, by enlisting the interest and support of students of Ceramics while still in schools, and by stimulating the spirit of ceramic research among them.

Granting of Charters. Application to form a Student Branch in any institution must be in writing, and signed by not less than five regularly enrolled students in good standing and endorsed by two or more members of the Society. The application must be filed and acted upon as provided for Local Sections in Section X, and may be suspended or revoked for cause in the same manner.

Legislative Powers. Student Branches shall have power to make their own rules and by-laws, except that they shall not pass any rule or by-law in conflict with the Rules of the Society.

Officers. The officers of Student Branches shall be a Chairman, a Secretary, a Councilor, and such others as the Student Branch may prescribe. The Chairman and Secretary shall be elected by the Student Branch, and their duties shall be such as usually pertain to these offices. The Councilor shall be an active member of the Society, appointed by the Board of Trustees to act in this capacity to the Student Branch. The duties of the Councilor shall be to advise the Student Branch in all matters pertaining to its relations to the Society and to make an annual report to the Society regarding the work and status of the Student Branch. The names of the Chairman, Secretary and Councilor for each Student Branch shall appear in the Roster of the Society in the Annual Volume of the Transactions.

Eligibility. Membership in a Student Branch shall be limited to the instructional force and regularly enrolled students of schools in which branches are located, subject to the rules of the school concerned governing outside activities of students.

Finances. Student Branches shall have power to fix their own dues and assessments, and as such shall pay no dues or initiation fees to the Socity. No student Branch shall have authority to incur debt in the name of the Society or for which the Society may become liable.

Relation to Parent Society. Members of a Student Branch as such shall pay no dues or initiation fees to the Society, but upon depositing a certificate of good standing from their Secretary may purchase from the Society its Transactions at the same rate as Associate members of the Society. This privilege shall cease when the student's connection with the school ceases, but members of a Student's Branch upon leaving school may at once become associate members of the Society, by depositing their certificates, making the proper application, and paying the regular initiation fee.

PUBLICATIONS

The following is a list of the volumes published by the Society and the prices at which they are for sale to the general public:

Description of volume.		Bound in paper.	Bound in cloth.
Vol. I.	1899, 110 pages	$4.00	$4.75
Vol. II.	1900, 278 pages	4.00	4.75
Vol. III.	1901, 238 pages	4.00	4.75
Vol. IV.	1902, 300 pages	4.00	4.75
Vol. V.	1903, 420 pages	5.00	5.75
Vol. VI.	1904, 278 pages	4.00	4.75
Vol. VII.	1905, 454 pages	4.00	4.75
Vol. VIII.	1906, 411 pages	4.00	4.75
Vol. IX.	1907, 808 pages	5.00	5.75
Vol. X.	1908, 582 pages	5.00	5.75
Vol. XI.	1909, 632 pages	5.50	6.25
Vol. XII.	1910, 882 pages	5.50	6.25
Vol. XIII.	1911, 837 pages	5.50	6.25
Vol. XIV.	1912, 888 pages	5.50	6.25
Vol. XV.	1913, 747 pages	5.50	6.25
Vol. XVI.	1914, 611 pages	5.50	6.25
Vol. XVII.	1915, 815 pages	*	6.25
Vol. XVIII.	1916, pages		6.25
Complete Set			$100.50

* Volume XVII and all following Volumes will be bound in cloth only.

The Board of Trustees established in the beginning a differential between members of the Society and others, in the matter of prices to be paid for copies of the Proceedings. This differential has been changed from time to time as volumes have grown scarcer and sales increased. The present arrangement, which will stand until March 1, 1915, allows a straight discount of 40 percent to members in good standing. Members in arrears are not furnished copies of the volume for the current year, nor allowed the discount for the older publication. Members are also not permitted to purchase more than one full set of publications at members' rates. The care and sale of the Publications has been transferred to the Publication Committee, which reserves the right to change the discount from time to time on any one volume or on all, as may be necessary.

The number of copies of the earlier volumes is not large, and members are advised to procure full sets as soon as possible. The prices will never be less and will certainly rise as time goes on.

In addition to the above volumes of the Transactions, the Society has also published the following books, which will be sold *net* at the prices listed to the public and members alike:

The Collected Writings of Dr. Hermann August Seger, Volume I. Contains (*a*) Treatises of a General Scientific Nature. (*b*) Essays Relating to Brick and Terra Cotta, Earthenware and Stoneware, and Refractory Wares. Pages, 552. Bound in cloth.....	$7.50
The Collected Writings of Dr. Hermann August Seger, Volume II. (*b*) Essays on White Ware and Porcelain. (*c*) Travels, Letters and Polemics. (*d*) Uncompleted Works and Extracts from the Archives of the Royal Porcelain Factory. Pages, 605. Bound in cloth.....	7.50
A Bibliography of Clays and the Ceramic Arts, by Dr. John C. Branner, 1906, 451 pages. Bound in cloth. Contains 6027 titles of works on Ceramic subjects.....	2.00

The above publications will be shipped at the consignee's expense, by express, to any address on receipt of the price. All checks or money orders should be made payable to the American Ceramic Society, and not to the Secretary or the Treasurer.

EDWARD ORTON, JR.,
Secretary.

Columbus, Ohio.

ANNUAL REPORT OF BOARD OF TRUSTEES.

CLEVELAND, OHIO, February 21st, 1916.

To the Members of the American Ceramic Society:

The Board of Trustees submit herewith its report for the year ending February 1st, 1916.

MEMBERSHIP

Again we record a moderate growth — 33 in all classes. Considering the entirely abnormal conditions abroad, and the reflex effect of this condition in America, the showing is a good one.

Status February 1st, 1915

Honorary members	4
Contributing members	2
Active members, resident	61
Active members, foreign	10
Associate members, resident	360
Associate members, foreign	46
Total	483

Accessions by election during the year

Active members, resident	5
Active members, foreign	1
Associate members, resident	60
Associate members, foreign	2
Total accessions	68

Losses during the fiscal year

Active members, resident, by death	1
Active members, resident, by resignation	2
Associate members, resident, by death	2
Associate members, resident, by resignation	7
Associate members, resident, by promotion to active	6
Associate members, resident, by non-payment of dues	13
Associate members, foreign	4
Total	35

Status February 1st, 1916.

Honorary members	4
Contributing members	2
Active members, resident	63
Active members, foreign	11
Associate members, resident	392
Associate members, foreign	44
Total	516
Net gain for the year	33

The analysis of a year ago on the membership question evidently bore fruit in some quarters. Everything that was said at that time might truthfully be repeated, but the chief point to remember is we should carry our propaganda actively forward wherever we can and enlarge the circle of men who might profit by the Society's work and are not doing so.

FINANCIAL STATEMENT

The money affairs of the Society are in good shape, as shown in the following statement:

RECEIPTS

Brought Forward from Preceding Year	$ 1,269.80	$ 1,269.80
Receipts from Dues for 1915.		
Active members	472.50	
Associate members	1,832.23	
Contibuting members	75.00	
Collections on arrears	182.00	
Payments made in advance	22.67	2,584.40
Receipts from Initiation Fees.		
From active members	40.00	
From associate members	290.00	330.00
Receipts from Sale of Publications.		
From Transactions	892 87	
From Branner Bibliography	1.81	
From Seger's Collected Writings	7.50	
From Cloth Bindings	111.17	
From Reprints	47.01	1,060.36
Receipts, miscellaneous	0.35	0.35
Total		$ 5,244.91

DISBURSEMENTS

Publication of Volume XVII.		
Stenographic report	$ 175.50	
Drawings, illustrations and engraving	415.24	
Printing, binding and casing	1,520.50	
Editor's salary, ($600), and expenses, ($30.85)	630.85	2,742.09
Paid expense of delegate to a meeting	1.95	
Paid for printing table of contents of first sixteen volumes	88.25	
Paid for Segers Collected Writings	10.47	
Paid for reprints sold to authors	130.25	230.92
Paid for Administration.		
Salary for assistant Secretary	300.00	
Extra clerk hire	211.75	
Postage, stationery and supplies	297.46	
Insurance and storage on volumes	73.50	
Telegraph and telephone	8.60	
Transportation of volumes by express and mail	70.14	
Binding and casing early volumes	0.42	
Refund for binding Volume XVII collected in advance	104.00	
Expenses at convention, including Section Q entertainment	146.05	1,211.92
Total		$ 4,184.93
Cash Balance in Bank		1,059.91
Total		$ 5,244.84

There has been an increase in net income of $249.50 for the year, but this has been more than eaten up by the increased costs for the printing of the Volume, and for every other department.

There have been three items this year of a new nature: First, the printing in circular form of Table of Contents of Volumes I to XVI inclusive, costing $88.25; second, the assumption by the Society of the cost of cloth binding for all members, which cost $104.00 for refunding the binding fees for the year already collected, and for the additional number of bindings to pay for; third, the assumption by the Society of the modest expenses of the smoker at Section Q. It seems to the Board

that all these extra expenses are well incurred. Two of them will be annually recurring expenses, unless the Society orders otherwise.

PUBLICATIONS

The new Volume No. XVII, made its appearance later than ever before. There was a variety of contributing factors to the delay. The Volume itself is an excellent one, and unusual pains and care have been expended in editing it. It is one of the largest volumes yet published, and its contents are perhaps more condensed than heretofore.

An inventory of the volumes on hand shows the following status:

NO. OF VOLUME	NO. REMAINING ON HAND FEBY. 1ST, 1915	RECEIVED SINCE	DISPOSAL OF BY SALE	EXCHANGES	NO. NOW ON HAND
I	152	...	8	...	144
II	112	...	10	...	102
III	149	...	8	...	141
IV	125	...	9	...	116
V	91	...	9	...	82
VI	119	...	8	...	111
VII	128	...	9	...	119
VIII	110	...	11	....	99
IX	74	...	12	...	62
X	103	...	8	...	95
XI	139	...	9	...	130
XII	94	...	13	...	81
XIII	196	...	15	...	181
XIV	109	...	10	...	99
XV	146	...	17	...	129
XVI	232	...	56	...	176
XVII	...	749	501	30	218
Branner Bibliography...	760	...	9	...	751
Total stock on hand					2,836

The Volumes on hand are well cared for, in the Fire Proof Storage and Warehouse Co., and are insured for the sum of $2,000.00.

SUMMER SESSION

A large and enthusiastic three day summer session was held in Western Pennsylvania, beginning at New Castle, and extending to Bessemer, Crescentdale, and ending at Beaver Falls, New Brighton and Rochester. The attendance was the largest ever had at one of these gatherings — about 110 in all.

The Plants visited were:

The Universal Sanitary Mfg. Co., New Castle,
The Shenango China Co., New Castle,
The Lehigh Portland Cement Co., New Castle,
The Bessemer Limestone Co., Bessemer,
The Crescent Portland Cement Co., Crescentdale,
The Armstrong Cork Co., Beaver Falls,
The Ingram-Richardson Co., Beaver Falls,
The Brighton Chemical Co., New Brighton,
The Beaver Valley Pot Co., New Brighton,
The H. C. Fry Glass Co., Rochester.

In addition, the Society was entertained evenings at the New Castle Country Club, and at the Beaver Falls Country Club, and at luncheon at the Universal Sanitary Mfg. Co., and the Bessemer Limestone Company. The evening at Beaver Falls witnessed the installation of the Beaver Section of the Society, the first local section to organize under the new provisions of the constitution.

STUDENT BRANCHES AND LOCAL SECTIONS

The year has witnessed the installation of two student branches and one Local Section, under the new provisions of the Constitution which were initiated at the Wheeling meeting and were passed upon by letter ballot in April, 1915. The first of these to organize was The Ohio State University Ceramic Society into the new form in May, 1915, with a charter membership of about 65. President Hice was present and delivered the address, welcoming the new organization into the fold, and explaining their opportunities and duties. Professor Arthur S. Watts was appointed Councillor to the new organization.

In October, 1915, the second Student Branch was established at the New York State School of Clayworking and Ceramics, taking over the local Ceramic Society and converting it into the New York State Student Branch, under the Councillorship of Professor Charles F. Binns. President Hice also officiated at this interesting occasion. There were twenty- two charter members.

The Beaver Ceramic Society of the Beaver Falls-New Brighton district, entered into new existence as the Beaver Section of the American Ceramic Society, on August 26th, 1915, at the Beaver Falls Country Club, before an audience of 50 or more members of the Society. The Beaver Section came in with a charter membership of about 35, and after disbanding the old organization elected its previous list of officers to serve in the new capacity. Mr. F. W. Walker was appointed Councillor for the Beaver Section. President Hice being President of the Beaver Section also, the Secretary, Professor Orton, delivered the principal address of installation to the new section. The organization seemed a very strong and influential one and likely to make its influence felt in the Society.

The foregoing accounts of the work of the Organization in its various departments, indicates that it is in a healthy, vigorous condition and carrying forward the propaganda for better knowledge and better practice in the ceramic industries in a effective manner.

For the Board of Trustees,

EDWARD ORTON, JR.,
Secretary.

IN MEMORIAM

IRA L. CONKLING

NAGATAKA FUJIYE

PAPERS AND DISCUSSIONS

(55)

PRESIDENTIAL ADDRESS.

THE FIRE CLAYS OF PENNSYLVANIA

BY RICHARD R. HICE

The importance and the value of the Pennsylvania fire clays are evidenced by the figures showing the production of fire clay refractories. In 1913 the State produced the equivalent of 361,548,000 clay fire brick, or 42.88 percent of the output of the United States. That this enormous output is not due, at least not entirely, to the proximity of the great centers of iron production, but rather to the quality of the output, is evidenced by the fact that the average price per thousand at the plants was $19.62, which was but 66 cents below the average price for the whole United States.

The figures of production do not include 214,734,000 front brick, largely made from fire clays, nor large quantities of clay paving brick made from the lower grade fire clays.

The industry is mainly confined to 18 counties, and of these Allegheny, Mercer, Berks, Washington and Lehigh obtain their clays from other fire brick centers. Almost the entire product of Beaver County and part of that of other counties is manufactured from the lower grade plastic clays of the Allegheny series. These facts explain the difference in value per thousand in some of the counties shown in Table I.

All the clays used belong to the Pennsylvanian portion of the Carboniferous rocks. Those found in the Pottsville are largely flint clay, while those of the Allegheny formation are prevailingly plastic, or semi-plastic.

The Pottsville clays are recognized as belonging to the Upper Mercer horizon, and are found underneath that coal bed. The Upper Mercer horizon belongs underneath the Homewood sand-

TABLE I — PRODUCTION OF CLAY FIRE BRICK IN PENNSYLVANIA 1913

COUNTIES	REPORTING PLANTS	QUANTITY	VALUE	PRICE PER THOUSAND
			dollars	*dollars*
Clearfield	7	101,141,000	2,209,062	21.84
Clinton	8	52,840,000	968,048	18.32
Allegheny	6	29,237,000	815,044	27.87
Cambria	5	34,432,000	690,830	20.06
Fayette	9	30,643,000	472,613	15.42
Westmoreland	5	20,577,000	395,407	19.22
Center	4	23,203,000	376,149	16.21
Beaver	6	21,754,000	250,432	11.51
*Elk, Armstrong, Clarion	5	20,938,000	334,506	15.97
*Mercer, Bedford, Somerset, Berks, Indiana	5	18,237,000	364,135	19.96
*Washington, Lehigh	4	1,771,000	41,621	23.50
Other Counties		5,775,000	176,947	30.64
		361,548,000	7,094,794	

* These counties are combined to avoid disclosing individual output.

stone, the massive top member of the Pottsville as found in the western portion of the State. The clays of the Allegheny formation are generally, but not always, found immediately beneath the several coal beds, and are practically co-extensive with the coals. The old idea that where a coal is of workable thickness the clay is thin, and where the clay is workable the coal is practically absent, has no foundation in fact.

A typical section of the lower portion of the Pennsylvanian rocks would be:

Conemaugh Mahoning sandstone

Allegheny ....
- Coal, Upper Freeport
- Clay, Upper Freeport
- Shales
- Limestone, Upper Freeport (Bolivar clay horizon)
- Shales
- Coal, Lower Freeport
- Clay, Lower Freeport
- Shales
- Limestone, Lower Freeport
- Shales and Sandstone
- Coal, Upper Kittanning
- Clay, Upper Kittanning
- Shales and Sandstone
- Coal, Middle Kittanning
- Clay, Middle Kittanning (some flint clay)
- Shales
- Coal, Lower Kittanning
- Clay, Lower Kittanning (some flint clay)
- Shales and Sandstone
- Limestone, Vanport
- Shales and Sandstone
- Coal, Clarion
- Clay, Clarion (some flint clay)
- Shales and Sandstone
- Coal, Brookville
- Clay, Brookville
- Shales

Pottsville .....
- Sandstone, Homewood
- Shales, Sandy
- Limestone, Upper Mercer
- Coal, Upper Mercer
- Clay and shales (flint clay)
- Shales
- Limestone, Lower Mercer
- Coal, Middle Mercer
- Clay, Middle Mercer
- Shales, Sandy
- Coal, Lower Mercer
- Clay, Lower Mercer
- Shales
- Sandstone, Conoquenessing

It is, of course, obvious that all of these strata are not everywhere present, and this is more true of the Pottsville than of the higher Allegheny rocks.

It is not my intention to try to speak of all the localities where good fire clays have been found, but simply to touch on a few of the "high spots", and in doing this I shall take up the subject by counties, rather than by the geological horizons of the several clays.

Clearfield County. As shown by the table Clearfield County produces in round figures 30 percent of the clay fire brick of the State. This is undoubtedly due, in part, to the quality of the clay found there, but it is in part due to the early development of the region and the reputation made for the product years ago. The users of fire brick dislike to make a change from a satisfactory article, they are in this line very conservative, and rightfully so. Since the early manufacturers have consistently maintained the high grade of their product, there is little inducement for a user of brick to make a change, with the possibility of failure, even if tests show another brick of equal merit. The average user of fire brick takes little stock in tests.

The exposed rocks in Clearfield County range from the Mauch Chunk of Mississippian age up to the Conemaugh of Pennsylvanian age. The Mercer division of the Pottsville ranges

from 10 to 40 feet in thickness, and the overlying Homewood from 50 to 75 feet. All the recognized coals of the Allegheny series are present, and each has its accompanying underclay.

The county is crossed from northeast to southwest by two well developed anticlines, and it is these folds which bring up the Mercer horizon to daylight so that it can be worked without shafting. The actual workings have been largely determined by the proximity of transportation lines, and undoubtedly large areas of good clays are undeveloped.

The largest area of Pottsville rocks extends in a wedge shape from the northern boundary of the County towards the southwest near Glendale. This has been brought to light by the Chestnut Ridge anticline, and is, without doubt, a likely area for Mercer clays, but it is without transportation facilities, and the details of the geology are little known. As there is no coal or other mineral of value in the area, the building of railroad lines seems remote. In the other portions of the county, the cutting of the valleys has often exposed the Pottsville, and with the nearness of the lines of transportation the clays have been developed.

Much of the County is marked by faulting, and most clay mines have them. The displacements range from a few inches to more than 100 feet, and some are fault zones rather than simple faults. There are occasional horizontal thrusts.

In addition to the Mercer horizon the Clarion, Middle Kittanning, and Lower Freeport clays sometimes carry a good portion of flint clay. As a whole, however, these higher clays in Clearfield county are plastic and of lower grade than the Mercer clays. The flint portion is often fractured, and the fractures filled with impurities — often they carry sandy inclusions. While the workable deposits of the Mercer clays are lenticular and often quite limited in extent, it is rather rare for the flint portions of the higher clays to be of workable dimensions, and they are only taken out in connection with the more regular plastic portions of the beds.

The Mercer clays of Clearfield county are quite regular in chemical composition. The silica ranges from 42 to 46 percent, the alumina from 36 to 39 percent. The statement often made

TABLE II — ANALYSES OF MERCER FLINT CLAYS FROM CLEARFIELD COUNTY, PENNSYLVANIA

	1	2	3	4	5	6	7	8
SiO_2	42.700	43.350	44.550	46.250	45.450	45.230	42.94	42.16
Al_2O_3	37.600	37.550	39.000	37.500	36.125	38.030	38.37	38.74
Fe_2O_3	3.385	2.145	1.440	1.935	2.275	1.980	1.11	1.34
TiO_2	2.500	2.825	1.700				1.88	2.08
CaO	.112	.084	.028	.168	.168	.163	.16	trace
MgO	.270	.234	.072	.126	.342	.237	trace	trace
Na_2O) K_2O (.....	.730	.235	.530	1.115	1.290	.830	1.25	2.22
H_2O	13.840	14.170	13.660	13.540	13.730	13.605	14.30	13.54

1. Two and one-half miles west of Blue Ball, vicinity of Morgan Run, Dark Grey Block clay. *Penna. Sec. Geol. Surv. H7*, p. 170. S. A. Ford, Analyst.
2. Two and one-half miles west of Blue Ball, vicinity of Morgan Run, Dark Grey Block.
3. Two and one-half miles west of Blue Ball, vicinity of Morgan Run, Light Grey Block.
4. Woodland, *loc. cit.* p. 171, McCreath, Analyst.
5. Woodland, *loc. cit.* p. 172, S. A. Ford, Analyst.
6. Woodland, *loc. cit.* p. 173, McCreath, Analyst.
7. Lumber City, Enrique Touceda, Analyst. (Good Fire Brick Co.)
8. Lumber City, Enrique Touceda, Analyst. (Good Fire Brick Co.)

that the quality of these clays is in inverse ratio to the Fe_2O_3 content does not seem to agree with known analyses. The analyses in Table II may be taken as typical of the Clearfield Mercer flint clays, and are given for whatever they are worth.

The important areas now being worked are the Woodland, Morgan Run, Anderson Creek, Lumber City and Clearfield Creek. While there are differences in the clays from these sections, yet on the whole the clays of Clearfield county produce brick of a remarkable uniformity in quality.

In addition to the clay used at the several manufacturing plants of the county, large quantities are shipped, some to other plants and a part for use as mortar clays. The last are from the plastic portions of the beds.

Clinton County. The second producing county of Pennsylvania is Clinton, which lies northeast of Center county. The portion of the county in which we are interested is entirely made up of rocks of Pennsylvanian and Mississippian age. A generalized section of the exposed rocks would be:

			Feet
Carboniferous	Allegheny	Coal, Middle Kittanning	2
		Shales	45
		Coal, Lower Kittanning	4
		Clay, Lower Kittanning	3
		Shales, sandy	
		Coal, Clarion	1
		Sandstone, Clarion	25
		Coal, Brookville	3
		Clay, Brookville	2
	Pottsville	Sandstone, Homewood	100
		Mercer	30
		Sandstone Conequenessing	100 to 200
	Mississippian	Mauch Chunk	
		Pocono	
Devonian			

The Allegheny series is only found in the tops of the higher hills in two synclinal basins, and the coals are practically of no importance. The Mercer clays are found in the same areas, where they are exposed in the valleys. There would seem to be no reason why they will not be found farther from the present lines of transportation. They are more irregular as regards deposition, and the internal structure of the bed is also more irregular than the same horizon in Clearfield County. This is no doubt due to their position in the great Appalachian trough, the conditions here being more affected by changes during the time of deposition than nearer the center of the trough.

The Mercer clays are found in two belts across the county. One extends from near North Bend on the Susquehanna river southwesterly, through the Woodland, Morgan's Run and Clearfield Creek districts of Clearfield County. It is quite narrow, ranging from three to six miles in width. Its southern boundary is the Susquehanna River. The second belt extends from near Farrandsville southwesterly across Center County.

The Mercer clays of this county are markedly superior to those found in the Allegheny series, this difference being more marked than farther westward.

The following analysis by McCreath will serve as typical of the Mercer clays of the Farrandsville region.

Analysis of Flint Clay from near Farrandsville:

SiO_2	42.440
Al_2O_3	36.685
Fe_2O_3	2.128
TiO_2	4.000
CaO	.200
MgO	.276
K_2O,Na_2O	.718
H_2SO_4	.820
H_2O	13.370
	100.637

The high percentage of TiO_2 is to be noted in connection with analysis.

Allegheny County. While the output of Allegheny County ranks third in value, yet the raw materials used are brought from outside the County. One of the early inducements for making fire brick here was the quantity of burnt clay obtained from the old used brick. To use this, however, it was necessary to bring large quantities of raw clays, and the industry has continued. The average price per thousand of the Allegheny County brick shows the effect of adding the freight to the clay, the average price being $27.87, or more than $8.00 above the average price in the State. A part of the difference in value may be due to the quantity of special shapes made.

Cambria County. The quality of the brick made in Cambria County is attested by the price. A large part of the brick made are used locally, and the fuel cost is low. As an offset to this, most of the clay is transported some distance, some from outside of the County.

The rocks exposed in Cambria County not only include the Allegheny and Pottsville, but a thickness of some 850 feet of Conemaugh above the Allegheny, and the Mississippian rocks below the Pottsville, with some Devonian. The materials used for fire brick include not only those of the Mercer horizon, but the clays of the Allegheny formation.

A clay of some interest has been reported at 50-75 feet above the Upper Freeport coal. This is a clay with a fine flint structure and furnishes us with a fine example of the fact that all flint clays are not fire clays.

Partial analysis of "flint" clay from Cambria County:

SiO_2	50.3
Al_2O_3	21.3
Fe_2O_3	10.4
MgO	.61
CaO	.39
Na_2O	.18
K_2O	1.14
TiO_2	.90
Loss on ignition	12.00
	97.22

This is one of the cases where the iron content condemns, and is given as one of the cases where a chemical analysis shows a "flint" clay to be unfit for use.

In this County rather extensive use is made of the Lower Kittanning clay as a binder for the Mercer flint. This clay is not only mined for this purpose at some of the plants, but it is mined elsewhere in the county and shipped by rail to other manufacturing plants. This use indicates that this clay is a higher grade than usual.

The Clarion clay is mined in a small way and shipped. It has a thickness of about six feet.

The Mercer horizon occurs along the Conemaugh River at the western boundary of the County and keeps above water level for several miles. It outcrops along the Little Conemaugh from South Fork to Mineral Point and is seen at other points along that stream.

Along Clearfield Creek, the Mercer appears near Dysart and at Dean, and continues northeast along Laurel Run as far north as the head water of Sandy Run. Exposures are also found on Black Lick Creek from Wilbur to Nanty Glo.

It is noted in Cambria County that whenever the Upper Mercer coal is present there is plastic clay beneath, usually thin, pure and refractory. The general succession is coal, plastic clay, flint clay, Conoquenessing sandstone, in descending order.

It is claimed by some that the flint clay from near Dean is the hardest of the Pennsylvania clays. As it lies close to the mountains it would be of interest to know whether this is due to metamorphic action connected with the folding of the region.

Fayette County. It is to be noted the average price of the fire brick made in Fayette County in 1913 was but $15.42, being the lowest price with the exception of Beaver County. This is due to the development of the industry to supply coke oven brick. Such brick are relatively low grade and do not permit of heavy freight rates and therefore induce the use of local materials. This fact, together with cheap fuels, probably accounts for the low price prevailing. It is not meant there are no high grade brick made, but these appear to be

relatively few. Many of the brick are made by coke manufacturers, and this fact shows their use.

Westmoreland County. Many of the brick made in Westmoreland County are also for coke oven use, but the manufacture of a considerable quantity of better grade brick for other purposes, raised the average price. The materials used in Westmoreland County are the clays of the higher series, including the Bolivar horizon, and also some of the lower flint clays. A portion of the raw materials is brought from outside the county. It is impossible to give the details of production.

Center County. The clays of Center County belong to the several horizons of the Allegheny and the Mercer. The entire Allegheny formation is present in the northwest half of the county, with the Pottsville beneath. The area in which the Mercer has been found, either in outcrops or by drilling, is rather limited. At the southwest corner of the county the known belt is about four and one-half miles in width. It widens towards the northeast to about ten miles. This area is a part of the same region developed in Clearfield County to the southwest, and the bed has the same characteristics as there, carrying not only a good portion of flint clay but large quantities of plastic material of a high grade. There are several plants making front brick from the plastic portion of the Mercer. These are located in the northeastern portion of the county. To produce a good front brick, one that will keep its appearance after being laid in the wall, requires burning at a high temperature. If analyses at hand of the plastic clay at Sandy Ridge are typical, there is a much higher alkali content than in Clearfield County.

There is a clay shipment of about 40,000 tons from this county but this is offset by the shipment of clays from Clinton County to the plants in the northeastern part of the county.

Beaver County. The manufacture of fire brick in Beaver County is an old industry, although the older plants have passed out of existence. The brick now made in the county are entirely, or practically so at least, made of the clays of the Allegheny formation, the Lower Kittanning clay being used. The Clarion clay has been used to a slight extent, and it is probably a higher grade clay than the Lower Kittanning. It

is, however, not so easy of access, is not so regular in occurrence and generally thinner when present. The Lower Kittanning clay is not only used for fire brick but largely for front brick. The quality is generally considered quite uniform, but it is probable that there is quite a difference as regards fire resistance. At one place at least where it was formerly used, it is now known to carry considerable flint clay, not in lenses, or in any regular position in the bed, but scattered through the clay in small particles, not exceeding one-fourth inch in diameter. At one time the fire brick made at the plant where this clay was worked had a good reputation, due probably to this percentage of flint, which was not recognized at that time, so far as known.

TABLE III — ANALYSES OF SANDY RIDGE CLAYS, CENTER COUNTY, PENNSYLVANIA

	1	2	3
SiO_2	45.650	44.950	45.820
Al_2O_3	34.730	37.750	35.950
Fe_2O_3	3.546	2.700	3.330
CaO	.112	.302	.112
MgO	.619	.216	.573
K_2O, Na_2O	5.750	.985	4.130
H_2O	9.650	13.050	10.130
	100.057	99.953	100.045

1. Upper plastic clay, *Penna. Sec. Geol. Surv. T4*, p. 117.
2. Flint clay, *loc. cit.*
3. Lower, plastic clay, *loc. cit.*

The other clays of the Allegheny are not worked to any extent. At one time the Bolivar was mined and used in the manufacture of saggers at Beaver Falls.

Elk County. The exposed rocks of Elk County include the lower portion of the Conemaugh, the Allegheny formation, and the Pottsville, of the Pennsylvania system, the Mississippian system, and the top of the Devonian. The higher rocks are only found in the southeastern part of the county. The Pottsville are the dominant rocks.

The Lower Freeport horizon is worked in the Shawmut region as is also the Lower Kittanning. The Clarion clay is not as widely distributed as is the Clarion coal.

The Mercer horizon is cut by most of the streams of the county, but only two localities are known to carry the clay, at Benezette (now worked out) and at Daguscahonda. Doubtless drilling would show other localities.

Armstrong County. The exposed rocks are of Mississippian and Pennsylvanian age. The oldest rock is the Burgoon sandstone and the youngest immediately above the Ames limestone, near the middle of Conemaugh.

The Mercer horizon is exposed at a number of places, but the clay is apparently often absent, and probably no systematic effort has been made to discover it.

The Clarion clay is wide spread and has given this county its standing as a producer of front brick. It is not, however, used for fire brick except as a binder. Considerable of it is shipped for this purpose. The Lower Kittanning clay is only worked at Kittanning, although it is found widely distributed over the county. The Upper Freeport clay is not used to any great extent at present, and the Bolivar is not known, although the Upper Freeport limestone is reported.

Clarion County. The rocks of Clarion County extend from the Mahoning sandstone, the base of the Conemaugh, to the base of the Pocono, some 650 to 700 feet. The lower rocks, however, are only found in the deeper valleys.

The Mercer horizon outcrops along the Allegheny and Clarion rivers and Red Bank Creek, together with the lower reaches of the larger tributaries. The formation varies from nothing up to 40 feet in thickness. The internal structure also varies more than in the counties farther east, as should be expected from the conditions attending deposition. Occasionally two beds of coal are present — generally but one — and sometimes nothing but carbonaceous shales mark the coal horizon. The clay is irregular and often absent. There are two localities where the Mercer is mined. One along Red Bank Creek, near Climax and New Bethlehem, and the other six to ten miles north and east of Clarion. The clay from near Mayport, analyzed by the United States Geological Survey, gave the following result:

TABLE IV — ANALYSES OF CLAY FROM NEAR MAYPORT, CLARION COUNTY

	1	2
SiO_2	58.96	56.46
Al_2O_3	25.60	27.69
Fe_2O_3	3.32	2.55
MnO	.07	.09
CaO	.70	.42
MgO	.25	.28
SO_3	.07	.14
Na_2O	.15	.06
K_2O	.36	.52
H_2O	.80	1.40
Ignition loss	9.90	10.64
	100.18	100.25

1. Top clay.
2. Bottom clay.

Comparing these analyses with those from Clearfield County, we note the silica content is 13 percent greater, while the alumina content is 11 percent less, quite a difference, if the value of the clay is to be determined by a chemical analysis.

The Allegheny series is quite widely distributed, but the clays are not as satisfactory as the Mercer. Most of the flint and much of the plastic clay mined is shipped in the raw state to points outside the County.

Mercer County. The output of clay fire brick in Mercer County is from clays shipped in from other places.

Bedford County. The production of brick in Bedford County is from clay from the Mercer horizon, locally designated as the Mount Savage clay. The basin is limited in extent, being confined to a sharply defined arch and the cost of mining is probably greater in other Mercer localities.

Somerset County. The fire brick production of Somerset County is also confined to the Mount Savage district, both the Bedford and Somerset County plants being close to the dividing county line.

Indiana County. The most of Indiana County is made up of rocks of Conemaugh age, but the cutting by the streams of the anticlinal axes which parallel the mountains has exposed

some of the underlying strata. In spite of this the Mercer horizon thus exposed seems to have no value as a clay producer, and the importance of the County as a producer of fire brick in the past has been due to the Bolivar clay.

The most important clay at the present time in the county is the Clarion. This is extensively mined and used at the brick plants at and near Bolivar. The Lower and Middle Kittanning clays as also some from the Bolivar horizon are also used at these plants. The Lower Kittanning is also mined at Clymer, but not used for fire brick.

Neither the Bolivar nor the Upper Freeport clays are mined to the extent they were in the past, but there is probably a large quantity of Bolivar clay which is of great value, although the best at the type locality has been exhausted.

The Bolivar must not be confused with the Upper Freeport. Stevenson in 1877 distinctly pointed out the horizon of both these clays, giving the following section east of Lockport.

	feet	*inches*
Mahoning sandstone	45	
Clay	1	
Upper Freeport coal	7	5
Clay	2	
Sandstone	1	
Clay	10	
Upper Freeport limestone	8	

Near Bolivar, Stevenson gives the following succession:

		feet	*inches*
1.	Mahoning sandstone	20	
2.	Clay		3 to 6
3.	Upper Freeport coal	8	9
4.	Fire clay, drab	3	
5.	Sandstone	2	
6.	Clay with iron ore	3	
7.	Coal		4
8.	Clay	30	

Stevenson says "the fire clay immediately below the coal is occasionally digged, but it seems to be too irregular to be mined

with profit. The little coal bed number 7 is persistent for some distance.

"Number 8 contains the non plastic Bolivar fire clay, which is of some irregular occurrence, as it varies from 3 to 12 feet in thickness and in some cases is wholly wanting."

Platt, (1877) gives the following section on the west side of the Chestnut Ridge.

	feet	*inches*
Coal, Upper Freeport	3	6
Clay with bands of sandstone	15	
Coal	1	
Clay	3	
Limestone, Upper Freeport	5	
Clay and iron ore	5	

On Yellow Creek the following section is found:

	feet	*inches*
Mahoning sandstone		
Upper Freeport Coal	6	
Clay	8	
Iron ore	3	2
Clay	3	
Coal	1	
Clay	4	
Clay, thin ore seams	3	
Limestone	1	

Unquestionably the loose use of the terms Bolivar and Upper Freeport as applied to these clays has led to confusion. The sections given which might be supplemented from many other localities, show clearly the presence of two distinct beds, and the thin coal at some of the localities must not be over-looked, or the fact that it has been noted at points quite widely separated. This clay is perhaps more lenticular than the Mercer.

The other counties where fire brick are made in any quantity are Washington and Lehigh. In both these counties high grade clay is brought in from outside sources, and the high freight rate on the raw materials is reflected in the price of the finished brick, the average for these counties being $23.50 per thousand.

NOTE ON THE METHODS FOR SATURATING CLAY TRIALS FOR ABSORPTION AND POROSITY DETERMINATIONS

BY M. F. BEECHER

In looking for a rapid and reliable method for saturating clay trials for the determination of absorption and porosity, the available data upon the relative merits of different methods were found to be very meager, and since the work in hand necessitated the determination of absorption upon some eight hundred specimens, it was advisable to make a few preliminary tests for the purpose of providing data from which to make a selection. So in the present instance there was no intention of making an extensive investigation into the matter of absorption methods but just a quick and reasonably comprehensive survey to suit the needs of the moment. The results obtained presented some interesting comparisons and they are here reproduced with the hope that they may be of use to others.

Most of the methods that have been used for saturating test specimens are given in more or less detail by the following writers in these Transactions:

Wheeler, Volume VIII, page 156.
Purdy and Moore, Volume IX, page 210.
Ries, Volume IX, page 693.
Bleininger and Moore, Volume X, page 303.
Brown, Volume XII, page 269.
Bleininger and Brown, Volume XII, page 346.
Bleininger, Volume XII, page 566.
Worcester, Volume XII, page 831.
Weelans and Ashley, Volume XIII, page 103.
Bleininger and Boys, Volume XIII, page 388.
Ogden, Volume XIII, page 400.
Kramm, Volume XIII, page 692.
Brown, Volume XIV, page 296.
Bleininger, Volume XV, page 73.

O'Connor, Volume XV, page 254.
Montgomery, Volume XV, page 610.
Orton, Volume XVI, page 511.
Kerr, Montgomery and Fulton, Volume XVII, page 97.
Beecher, Volume XVII, page 106.

The several methods pursued in the present instance cover substantially those outlined in the foregoing references. Some data on the rate of absorption are not included because of their incompleteness.

The Specimens Used. The bodies were all grogged clay. The grog varied in size from 30 to 120 mesh and in amount from 20 to 70 percent. The specimens measured about 2 in. by 4 in. and were ¾ in. thick. They were selected so as to represent the whole range of porosities to be encountered in the work at hand.

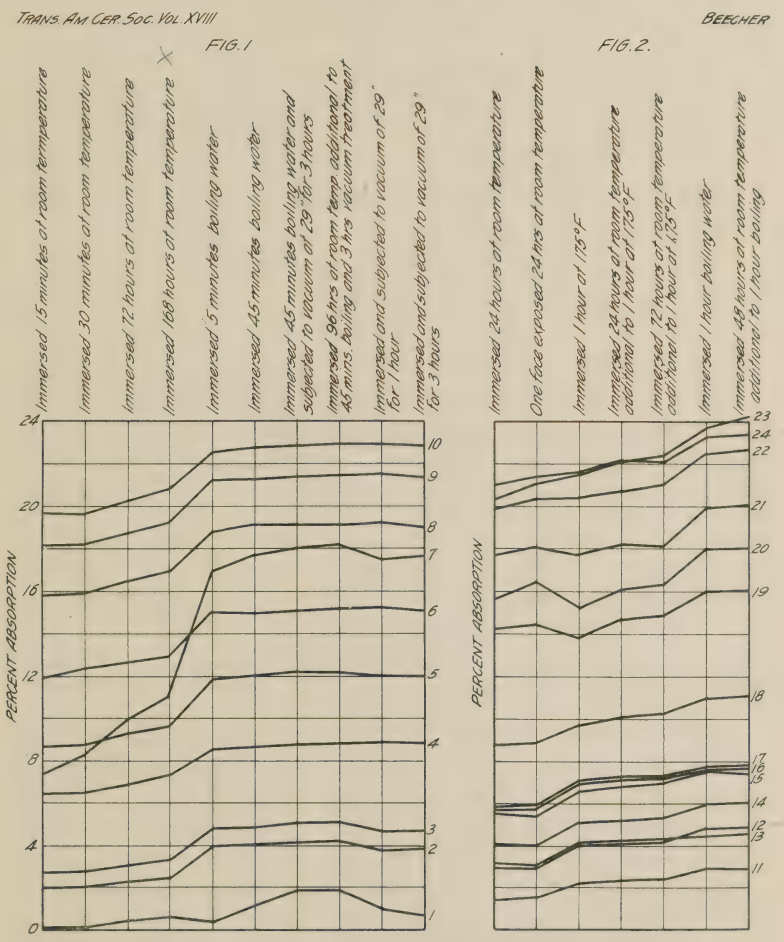
Laboratory Methods. Although the same specimens were used for the whole series of tests, the dry weights were checked before each test. At the conclusion of the series, the absorption was again obtained by the method first employed, to insure that the actual porosity of the specimens had not been changed either by deposition from, or solution by, the water used. Tap water was used throughout the work.

No refinements other than ordinary laboratory practice demands, were introduced. All weights were determined to a tenth of a gram. The saturated specimen was dried with a soft towel without rubbing before being weighed. When specimens were "immersed in boiling water" the water was boiling when the specimens were immersed and kept boiling vigorously until the specified time was up.

Discussion of Results. Tabulated and graphical presentation of the data obtained may be found in the tables and figures accompanying. Since the number of specimens here considered is so small and the range in structure and other characteristics so narrow, the deductions that may be made are limited accordingly.

That thorough saturation cannot be obtained by immersing for any reasonable period at room temperature is evident from Figure 1 where the curves show the saturation to be very in-

complete even after a seven day soaking period. It is also apparent that the rate of saturation under these conditions is greater for those specimens having high absorption; i. e., the



bodies with high absorption reach their highest degree of saturation by this method, in a less time than those with low absorption. This is apparent from the "slope" of the curves. The one exception is in the case of specimen No. 7. The evidence

of most importance, however, concerning the reliability of porosity and absorption values calculated from saturation by immersion at room temperature is that presented by curve No. 7 of Figure 1 and curves No. 12 and No. 24 of Figure 2. These show that such values are liable to be very misleading.

In the method of saturation by immersion in boiling water, the rate of saturation does not vary in the order of absorptions. Specimens No. 1 and No. 7 of Table I are the two striking examples. The boiling treatment, however, shows a decided improvement in the degree of saturation over the room temperature method.

Immersion in a vacuum showed slightly lower results on the average than the boiling treatment.

The highest results were obtained by a combination of the boiling treatment with the vacuum and a long soaking period. Though the effectiveness of this soaking period after boiling and vacuum treatment may not seem logical, in Table II and Figure 2 there is again shown an increase in degree of saturation due to a soaking period following a one hour boiling treatment.

Because of the similarity of all the bodies used it is not possible to discuss the possible relation between body structure and saturation behavior. The behavior of specimen No. 7, however, gives promise that interesting results might be expected from such a study.

Conclusions. From the data presented it was decided that for ordinary laboratory practice where the time element as well as accuracy is a factor, and where the results are for comparison only within a given study, saturation by boiling for forty-five minutes to one hour is sufficient for porosity and absorption determinations. Treatment with a vacuum alone does not offer better results.

Where absolute accuracy is the important consideration a combination of boiling with the vacuum treatment and a soaking period is the most effective means of saturation. Whether the soaking period first, rather than last, would give higher results, was not determined, neither was the efficiency of the vacuum

TABLE 1—PERCENT ABSORPTION BY DIFFERENT METHODS OF SATURATION

SPECIMEN NUMBER	WEIGHT IN GRAMS	IMMERSED 15 MIN. AT ROOM TEMP.	IMMERSED 30 MIN. AT ROOM TEMP.	IMMERSED 72 HRS. AT ROOM TEMP.	IMMERSED 168 HRS. AT ROOM TEMP.	IMMERSED 5 MIN. IN BOILING WATER	IMMERSED 45 MIN. IN BOILING WATER	IMMERSED 45 MIN. IN BOILING WATER AND SUBJECTED TO A VACUUM OF 29″ FOR 3 HRS.	IMMERSED 96 HRS. AT ROOM TEMP. ADDITIONAL TO 45 MIN. BOILING AND 3 HRS. VACUUM TREATMENT	IMMERSED AND SUBJECTED TO A VACUUM OF 29″ FOR 1 HR.	IMMERSED AND SUBJECTED TO A VACUUM OF 29″ FOR 3 HRS.
1	210.3	.10	.10	.48	.57	.38	1.19	1.80	1.80	.90	.62
2	217.5	2.02	2.02	2.25	2.48	3.92	4.00	4.08	4.14	3.68	3.82
3	198.5	2.72	2.72	3.02	3.27	4.74	4.83	4.98	5.03	4.58	4.68
4	292.3	6.43	6.53	6.74	7.35	8.48	8.62	8.86	8.86	8.82	8.79
5	204.1	8.67	8.72	9.30	9.75	11.86	12.04	12.19	12.19	12.00	12.04
6	270.5	11.93	12.31	12.65	12.95	15.00	14.95	15.12	15.19	15.22	15.05
7	220.2	7.35	8.22	9.89	10.98	16.92	17.69	18.01	18.19	17.51	17.65
8	221.0	15.79	15.85	16.51	17.05	18.80	19.13	19.13	19.09	19.23	19.05
9	212.1	18.13	18.18	18.80	19.31	21.25	21.28	21.40	21.42	21.49	21.35
10	218.9	19.65	19.65	20.27	20.80	22.62	22.73	22.86	22.87	22.91	22.86

TABLE II — PERCENT ABSORPTION BY DIFFERENT METHODS OF SATURATION

SPECIMEN NUMBER	WEIGHT IN GRAMS	IMMERSED 24 HRS. AT ROOM TEMP.	ON FACE EXPOSED 24 HRS. AT ROOM TEMP.	IMMERSED 1 HR. AT 175° F.	IMMERSED 24 HRS. AT ROOM TEMP. ADDITIONAL TO 1 HR. AT 175° F.	IMMERSED 72 HRS. AT ROOM TEMP. ADDITIONAL TO 1 HR. AT 175° F.	IMMERSED 1 HR. IN BOILING WATER	IMMERSED 48 HRS. AT ROOM TEMP. ADDITIONAL TO 1 HR. BOILING
11	216.9	1.38	1.47	2.21	2.35	2.40	2.86	2.90
12	208.2	2.93	2.88	3.99	4.13	4.22	4.80	4.89
13	208.4	3.07	2.93	4.08	4.22	4.27	4.46	4.56
14	198.0	4.09	3.99	5.00	5.15	5.31	5.96	6.06
15	218.1	5.50	5.41	6.55	6.78	6.92	7.52	7.56
16	209.8	5.71	5.72	6.91	7.11	7.15	7.63	7.72
17	216.6	5.81	5.91	6.97	7.20	7.29	7.66	7.75
18	206.8	8.70	8.85	9.77	10.07	10.20	10.98	11.12
19	211.9	14.25	14.54	13.78	14.63	14.93	16.00	16.00
20	230.8	15.62	16.47	15.29	16.05	16.29	17.98	18.05
21	213.0	17.70	18.12	17.84	18.21	18.12	19.97	20.15
22	209.4	19.90	20.41	20.41	20.70	21.02	22.55	22.66
23	185.0	20.32	21.08	21.41	22.05	22.33	23.68	24.28
24	209.0	21.05	21.36	21.60	22.10	22.10	23.30	23.40

treatment, described in an earlier volume,[1] in which the air is first exhausted from the specimen and then boiling water slowly admitted to cover it.

The method of partial immersion has little to commend it except in those cases where the specimens are too large to boil or submit to a vacuum, or where the purpose of the investigation demands such treatment in order to approximate certain conditions of use.

[1] Weelans and Ashley, Volume XIII, page 103.

PRACTICAL NOTES UPON THE MANUFACTURE OF FIRE BRICK SHAPES

BY H. L. LONGENECKER

Considerable data has now been published upon various methods of testing fire clays and fire brick, and there is still much to be learned in this direction. Very little has been written upon the subject of the mechanical details involved in the manufacture of fire brick and fire brick shapes, although upon this knowledge depends the success or failure of each fire brick plant. Many of the points in this paper are familiar to some fire brick manufacturers, but from observations in different plants I know that the information is not common knowledge.

There are probably few people besides fire brick manufacturers who realize the many different and difficult shapes into which fire clay is now worked and the ever-tightening specifications for this class of goods. As an example of how elastic specifications are in some parts of the country, standard fire brick, ordinarily figured as weighing seven pounds and measuring 9 in. by 4½ in. by 2½ in., are regularly being made from ⅛ in. to ¼ in. scant in all dimensions and as much as one-half pound underweight. Several progressive manufacturers are making full size, full weight brick and are winning large orders by demonstrating to buyers the greater cubical wall content per thousand brick which they give. Shape brick generally fit steel or cast iron frame work and cannot vary as brick which are laid in clay or mortar.

Specifications for shape brick are daily becoming more rigid. For important work, size specifications are now written as close as one-eighth inch total variation per lineal foot and one-quarter inch variation specifications are rigidly enforced even to 6 in. dimensions. Some manufacturers have perfected their methods sufficiently to meet these and other specifications

with small percentage loss and are getting quality prices. Special mixtures enter very largely into this business. The fire brick shape business represents the cream of this industry. Prices for standard brick are fixed by competitors, but quality alone largely determines the profit on shapes.

The use of special blocks for boiler furnace arches, rear arches for return tubular boilers, etc., has been made common by advertising. Special shapes are also used extensively for locomotive arches, locomotive oil furnaces, suspended flat arches for stokers, bridge wall blocks, cement kiln blocks, gas retorts, coke oven blocks, ladle blocks for pouring large and intricate castings, in fact are being more and more used in every heat working industry.

The problem of selecting the right mixture of clay to give best results in each service is one, the solution of which frequently wins or loses large orders. Installation costs and inconvenience generally exceed the cost of brick. The user will always pay more for the brick which gives the best service and he will stick tenaciously to the manufacturer who produces it. Some shape brick must be high in alumina and dense to resist slags, others must endure extreme variations in temperature, high temperatures, soaking heats, and many combinations and variations of these conditions. The proper mixture of clay will invariably give two, three or more times the life of other mixtures and densities of high quality clays, not suited to the service conditions.

Of the two varieties of fire clays, the hard or flint clay is generally purer and more refractory than the plastic clay. The majority of plants having their own mines or pits, however, possess a larger amount of the cheaper or plastic clay which encourages its excessive use.

Methods of Mixing. The common method of mixing fireclay is by shovelfuls and wheelbarrow loads. Where small variations in shrinkage are desired, this is a very crude way, although it sometimes produces good results. I know of an instance the most convenient pile regardless of the mix specified. A where the laborers wheeled the clay used on wet days from better method is the use of partitioned cars or trucks which are

filled either from overhead bins or by shovel. Where each apartment of such a partitioned car is filled level full by the same men continuously, variations in shrinkage are small.

Mixtures. For making the cheaper shapes, kiln floor blocks, arch blocks, etc., either plastic clay alone is used or sometimes about 80 percent plastic and 20 percent bat grog. Some plastic clays of fair refractory quality have a shrinkage as high as 1½ inches per lineal foot. The addition of 20 percent grog will reduce this to about 1¼ inches. For slightly higher quality ware, such as cement kiln blocks, a mixture of about 40 percent plastic, 40 percent flint and 20 percent grog is frequently used.

This has a shrinkage of about one inch per lineal foot. More refractory mixtures involve the use of more flint clay and grog.

The content of plastic clay can be reduced to a minimum by grinding to about 40 mesh. A high quality mixture having a shrinkage of ¾ to ½ inch per lineal foot consists of 50 percent to 60 percent hard clay, 15 percent to 25 percent plastic clay and 25 percent calcined hard clay. I know of one manufacturer who is making high quality shapes and brick of pure flint clay and grog only. Where minimum variation in dimensions and freedom from warping are desired, as high as 50 percent grog is used. This mixture has practically no shrinkage. The condition and quality of the grog is a large factor in producing high quality ware. Some manufacturers depend entirely upon bats, others make plastic clay into dobies and burn them between the bags of their kilns, but better results are obtained with the use of calcined flint clay. Calcined flint clay grog is sharp and makes a perfect bond with the plastic clay. The use of bats for grog sometimes reduces the refractoriness of the brick. Brick made with bats frequently show hair line cracks, indicating imperfect bonding. Such brick will not give the same length of service as perfectly bonded brick in withstanding variations in temperature, and they are not so impervious to slags.

Methods of Grinding and Tempering. Grog is always passed through a crusher before use, and the clays are sometimes passed through a dry pan and screened. Where this is done, the time required to temper is reduced. Common practice

is to grind and temper the clays together in a wet pan, leaving the amount of water to be added and fineness of grind entirely to the judgment and "feel" of the pan-tender. This is a very delicate point. Molders working on a piece work basis are seldom required to work ten hours and there is, therefore, a tendency to rush through, even to the extent of rushing the pan-tender. Clays require a definite time to temper, even though a molder may be rushing and out of clay, and it is a good idea for the pan-tenders to work by the hour. The time after the molders have gone can be utilized in stocking tempered clay for the next day. Stocked clay is undoubtedly of a more even temper, and it is an excellent arrangement for the molders to work always from clay which has been stocked several days.

Molding. Molding is a trade which takes several months at least to learn, and it would be difficult to describe all of the tricks and sleights involved. The manner in which the mold is made is very important, although the molder has little to do with it. For the more complicated shapes, best results are obtained by using two or more loose sides in the mold box. These loose sides principally decrease the distortion caused by removing the mold from the shape. It is of course impossible to mold many shapes without the use of loose sides. These loose sides have considerable draft or taper in the mold box, so that by the use of properly placed tapping holes, they come from it easily. Removing loose sides from the shape is a matter of peeling or sliding that requires considerable skill.

Different manufacturers and different markets, differ as to the lubricant used. Generally fine white, quartz sand is best. The use of river sand, or clayey sand, almost always results in sand rolls and folds. The use of oil causes oil checks and cracks and intensifies the fire-flash, so that oil molded blocks are generally distinguishable by their disagreeable color and fine oil checks. Oil checks and cracks can be largely overcome by using lard oil or a pure oil which volatilizes at a low temperature. The use of sand recovered from kiln flues will invariably discolor the brick. This sand had better be used by the setters. Where loose sides are used and where the "shape" is deep enough to justify, it is well to allow from $\frac{1}{8}$ in. to $\frac{1}{4}$ in. more shrinkage

on the bottom of the mold than on the top. This helps to take care of settle on the drying floor. Another good idea used with loose sides, is an arrow, imprinted in the brick, to indicate "way out of mold." This permits reversal of the block in setting to eliminate still further any distortion due to pull of the mold, or settle.

The mold box must be made of hard wood in order to resist the knocks of the molder's mallet and general rough usage, but the lining is generally of a softer wood, to prevent warping. Wear on the outside of the mold box is largely taken care of by "ironing it" *i. e.,* iron strips and bolts. The loose sides of the mold are banded, and a certain amount of wear can be compensated for by increasing the thickness of the shims or lugs on the backs of the loose sides. For cutting stiff tempered clay or for a wide mold, an excellent bow can be made similar to a wide hacksaw bow, with wing-nut tightening device. In molding shapes, the molder rolls and wedges, upon his molding table, a lump of a little more than sufficient clay to fill his mold. This clay lump or "walk" is worked without sand until it is free from air spaces and thoroughly welded together, its final form being a blunt wedge about the length of a molder's fore arms. Some molders then sand the clay lump very lightly. In the meanwhile, the molder's helper has cleaned, sanded and assembled the mold in a convenient position upon the floor. The molder grasps the "walk" with his hands and forearms and casts it into the mold. Molder and helper grasp the mold, and raise and drop it upon the molding block, from four to eight times. This block generally has a tree-stump for a base and a dry-pan breaker-plate for a top. The surplus clay is cut from the top of the mold with a bow wire, and the cap laid, cut side up, upon the molding table. It is much easier to avoid sand rolls if the next "walk" is built upon this cap. After the cap is removed the wire roughened surface of the shape is smoothed with a wooden spatula or "slicker", previously dipped in water. The top of the shape in the mold, is sprinkled with sand, the pallet board laid upon it and the mold is overturned upon the pallet. The loose sides are tapped free, the mold box lifted, and the loose sides slipped from the "shape". The "shape" is then carried upon the pallet

to its place on the drying floor. If the shape is particularly difficult, it is carried in the mold to its place on the drying floor and removed there. The shape is left on the drying floor, covered or without cover until it is leather hard, when the molder or "shape dresser" goes over it with a small trowel, trimming away fins. A mallet, board, and squares are used to flatten or round bulges and rough spots left from trimming fins, and to correct the block if it has settled out of angle or square. The mallet must be used with delicacy and judgment for a careless or incompetent "shape dresser" may do more harm than good.

Drying Cracks and Warpage. Drying cracks generally form at some indentation in the shape. If this indentation is not deep, and the parts which it separates are of equal thickness, a small wad placed in this cavity during drying will generally prevent this crack. The practice of filling a depression with damp sand is not always successful, for the reason that the sand does not dry at the same rate as the clay. Where the thin parts of the shape are of different thickness, the drying of the thin part or of the entire shape may be retarded with dampened cloths.

Warpage is frequently caused by warped pallets, sometimes new and made from unseasoned lumber. Where warpage is due to other causes it is nearly always toward the cut side of the "shape". Thin flat tile, 3 in. by 12 in. by 24 in. etc., are frequently subject to warpage. Sometimes this can be overcome by turning the tile when leather hard and slightly roughing the uncut side. The more nearly perfect the support of the shape upon the drying floor, and the lower the gravity center of the shape, the less will be distortion and settle in drying. The pallet should support every large angular face and groove on or in the under side of the shape.

Setting. Common practice is for the "green-brick wheelers" to convey the bricks and shapes to the "kiln setters" upon wheelbarrows. Sometimes three-wheeled trucks are used.

These wheelbarrows or trucks should be of the spring type and well padded. Where one green shape is piled upon another on the wheelbarrow, a pad should be placed between them.

Fire brick are generally set in bungs, four deep with a two inch flue or chimney between each two bungs. Every fourth header up and across is offset to tie across the flue. Unless a flash ring four headers deep is set around the kiln, these flues should be set to within four stringers of the bag walls. Five headers on two stringers makes a good setting. Shapes should not be set lower than four feet from the top of the kiln. Where rectangular kilns are used, it is good practice to set a tie of burned brick (which will not shrink or work during the entire course of the burn) from wall to wall of the kiln as a level floor for the shapes. The more nearly the entire bearing surface of a brick can be supported, the less liable it is to kiln marking, distortion, burning cracks or broken corners. Fine screened quartz sand should be used plentifully and as few setting wads as possible. Here is where a level kiln floor pays for itself. Setting wads must be well sanded to prevent sticking. Both sides and tops of high quality shapes should be protected by cheaper ware from fire-flash. Fire cracks may often be prevented by careful setting and properly placed wads. Setting shape brick is an art, and the setter who will govern his setting by a study of burned results, should be financially encouraged. Shapes should not be set bone dry as it is then extremely difficult to avoid broken corners. They are in the upper part of the kiln where they will dry rapidly and carry less weight than any other part of the kiln.

Burning. It generally takes a day or two longer to burn a kiln containing shapes. A little more time must be taken in water smoking, and the kiln must be soaked a little longer at the end, in order that the heat may thoroughly penetrate the thicker shapes. The burner should know exactly what is in each kiln. I know of one or two yards burning fire-clay shapes to cone 15, where the doors are pulled down as soon as the last fire is placed, and apparently without damage to the ware either physically or in service. This is done more because of a shortage of kiln room than for any other reason. Slow cooling for the first two or three days after the kiln is off fire is a much surer method of obtaining first-class brick.

The use of low sulphur coal will frequently produce whiter, cleaner brick.

The use to which the shapes are put must largely influence the hardness of burn. Shape brick should have all the shrinkage burned out. Many fire brick manufacturers, and plant superintendents argue that a soft burned brick will give better results than a hard burned one in withstanding alternations of temperature. If the life can be burned out of a fire brick, or if it will vitrify at a lower temperature than that to which it is exposed in service it is not suitable to withstand temperature alternations in that service. The ability to withstand alternations of temperature is largely dependent upon thorough bonding and a porous structure at the highest temperature to which it is exposed. The information given in this paper largely composes what a few years ago was commonly regarded as the secret processes of an industry.

The demand for more serviceable refractories must be satisfied with an intelligent preparation of the materials now in use to meet each individual condition, rather than the discovery of new refractory substances.

CHEMICAL PORCELAIN

BY E. T. MONTGOMERY AND M. G. BABCOCK, ALFRED, N. Y.

The European war has caused a serious shortage of the chemical porcelain which we formerly obtained from Germany and Austria and a number of American manufacturers have recently turned their attention to the making and marketing of this class of ware.

We have tested at our laboratory, for several of the large jobbing houses, a great many varieties of European porcelain as well as some of the American-made ware recently put on the market, and we are therefore in a position to present some data and make some suggestions which may be of value to the manufacturer just entering this field.

Receptacles made in chemical porcelain shapes, but using china bodies and glazes will serve many useful purposes about a laboratory, but to be called chemical porcelain and to seriously compete with the European product, this ware must meet certain definite requirements.

These requirements are as follows: It must be able to stand rapid changes in temperature without fracture; it must be thin, vitreous and translucent; and the glaze must be very hard in order that in blasting neither the triangle nor the precipitate will stick to the glaze, also that it shall be most resistant to alkaline solutions

Deformation or Fusion Tests. Some deformation test data on porcelain bodies will be of interest at this point. These tests were made on small cones one inch high and one-fourth inch wide at the base, cut from pieces of finished ware by means of an abrasive wheel. Such a cone from a cone 10 porcelain body deformed at cone 15. The composition of this porcelain was: clay, 40; flint, 30; spar, 28; whiting, 2.

Cones from two different American-made chemical porcelains deformed at cones 18 and 20 respectively. All European

chemical porcelains so far tested have deformed at cone 25 or above. It would seem from these tests that the temperature of cone 25 in the deformation test differentiates very nicely between what may be called hard bodies and medium or soft bodies.

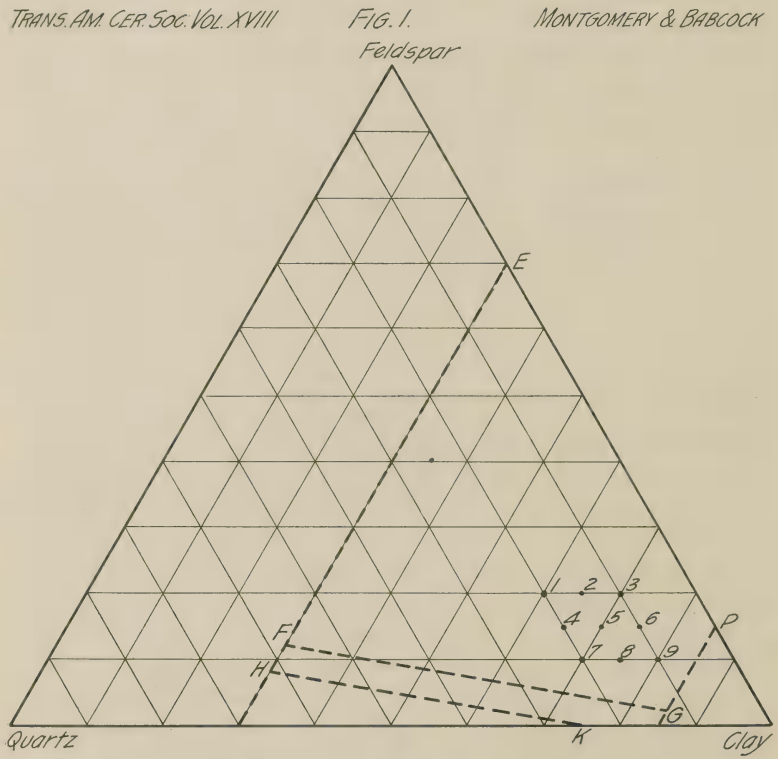
Rapid Heating and Cooling Tests. It should be pointed out that a deformation temperature of cone 25 or above is not enough in itself. This deformation temperature depends, of course, on the composition, and the burning temperature for the ware must be so adjusted to this composition that a strong vitreous product results which is capable of withstanding sudden heating and cooling. One European porcelain, which deformed considerably above cone 25, did not stand the rapid heating and cooling test quite so well as several other standard chemical porcelains which deform at or near cone 25. This was probably due to its not being fired to a sufficiently high temperature for its composition.

The rapid heating and cooling test which we have adopted as a standard is as follows: A small crucible, one and one-half inches in diameter at the top, is heated to a bright red in a blast-lamp flame, removed by metal tongs to a suitable support near at hand and a cold air blast from a compressed air line immediately turned on one side of the crucible only, until it is cold. This heating and cooling is repeated until the crucible breaks, if under ten times. A satisfactory chemical porcelain should stand ten successive heatings and coolings without crazing of the glaze or rupture of the body. European porcelain will on the average meet the requirements of this test. None of the American porcelains so far tested will even approach it. One make invariably broke on the first or second heating or cooling. The glaze was also so soft that it softened and blistered in the blast flame, sticking badly to the triangle.

Action of Acids and Alkalies. Porcelain glazes, being acid silicates, are not attacked to any appreciable extent by acids, but soft glazes especially are rapidly attacked by strong alkaline solutions.

Summation. We have seen that the deformation temperature of a chemical porcelain should be about cone 25; that the body, of a composition such that it will deform at this

temperature, must be fired sufficiently high to become vitreous and translucent, which means a firing temperature of about cone 16; and finally that the glaze must be a hard porcelain glaze maturing also at about cone 16. As both the body and the glaze must be high fired, the only commercially feasible process of



P E F G is Dr. Hecht's area of sound glazes at cone 16, when all of the clay substance in the body is from kaolin. This area is extended to P E H K when this clay substance is half from kaolin and half from plastic or ball clay. See page 260, Vol. XIII, *Trans. Amer. Cer. Soc.*

manufacture is the European low biscuit and high glost method. This gives us a high clay body, vitrified by heat and not by fluxes, bonded into a strong mass by sillimanite needles and free from undissolved silica as shown microscopically by Mr. A. A. Klein in another paper in this volume. It also gives us

a hard glaze which will neither blister nor stick in the blast lamp flame or be easily attacked by chemicals. Chemical porcelain does not differ materially from other hard porcelains except perhaps in that it is more aluminous and less siliceous. We know that Haviland china or porcelain table service is fired at cones 15 and 16 respectively in two different plants, while Royal Copenhagen porcelain is regularly burned at cone 18.

Experimental Work. Nine bodies of the composition shown in Figure 1 were ground in a ball mill with .5 percent of a mixture of sodium silicate and sodium carbonate and sufficient water to bring each to a good casting thickness. The clay content in each case was made up of fifteen parts of English ball clay with the balance equally divided between English china clay and Florida clay. Eureka flint and spar were used. Small crucibles, one and one-half inches in diameter across the top, and of uniform thickness, were cast in plaster molds. After drying and marking, these were fired, unglazed, to cone 16 and cooled slowly. All of the nine bodies developed good vitrification with fine translucency in the lower members of the series.

Testing. Three crucibles from each of the nine bodies were subjected to the standard rapid heating and cooling test with the following results: The bodies were found to be better able to stand this test as the clay content increased and the flint and spar contents decreased. It must be borne in mind, however, in connection with this statement that we are considering only vitrified bodies. Body No. 9 proved to be the best in the series, standing as good a test as Royal Berlin. Body Number 6 was but slightly inferior, and Number 8 was also good.

All of the crucibles from the first test which had stood ten consecutive heatings and coolings were now glazed with a hard porcelain glaze, again fired to cone 16 and tested again to see if the glaze had any effect in causing cracking or rupture. These glazed crucibles all stood ten more consecutive heatings and coolings without failure. It would seem therefore that at cone 16, body and glaze had so coalesced that there was perfect unity between them.

A fusion test was made on body Number 9 together with a piece of Royal Berlin porcelain. Both started to deform at cone 25, the Berlin bending slightly more than body Number 9.

Conclusion. It would seem, therefore, from the data presented, that in order to produce a satisfactory chemical porcelain we must have a hard porcelain body and glaze, fired together to at least cone 16, and sufficiently vitreous at this heat so that a test cone from the body will deform when the temperature is carried up to about cone 25.

THE PHYSICAL PROPERTIES OF SOME BALL CLAYS AND THEIR EFFECTS ON PORCELAIN BODIES

BY T. A. KLINEFELTER

The methods and data brought out in this paper are the result of an investigation to find a substitute for an English ball clay. All of the work was carried out to correspond as closely as possible to actual factory conditions, the drying, firing, etc., being done in the regular driers and kilns. All of this, of course, gives data which is only comparative in value. It was thought, however, that the method in particular would be of value to the practical man who must carry on tests under factory conditions, and that the data so obtained would be quite as valuable to him as if it were done in the laboratory. Indeed, the results should be more intelligible from his standpoint, since it is often necessary to translate, as it were, the laboratory results into factory results.

The steps followed were:

Clay sampled.
Sample ground.
Clay divided into a crude clay batch and into a standard body.
Both batches mixed with water and soaked.
Both blunged.
Body mixture screened.
Both dried to a state of plasticity.
Both worked up and wedged.
Both aged.
Worked again and cut into briquettes.
Briquettes marked.
Briquettes dried.
Drying data noted.
Fired.
Burning data noted.

For sampling, at least 50 pounds of clay should be at hand. This reduces the chance of error in case the clay does not run uniform in texture. Such clays as Tenn. No. 3, and several of the English clays would likely show quite varied results if the samples were taken from the tobacco pouch size samples so frequently sent out. A large sample of clay, also, gives something to check against in case an order is placed later on.

The entire sample was spread out and sampled in the regulation manner. About 1500 grams were then ground down in a mortar to such fineness that practically all would pass through an 18 to 20 mesh sieve.

One thousand grams of this were taken for crude clay tests. The remaining 500 were used in making up a standard body, 50 percent clay, 30 percent flint, 20 percent spar.

To both the crude clay and the body mixture, water was added, and the whole stirred and mixed with a spatula or stirring rod until a slip about the consistency of thick cream was formed.

At this point the clays began to show their individualities. The crude clay mixture, of course, brought out these peculiarities more than the body could. Some clays, such as Sant's English No. 1, Knowles' No. 12, the Kentucky Construction Co. Special and Black, slaked quickly and readily to the required consistency with about a third less water than such clays as Tenn. No. 3, and did not require nearly so much working. In fact the sticky, gummy clays would hold small gummy clots after the most persistent working.

The slips were not brought to a standard weight, as more water had to be added later on anyhow in the case of some of the crude clays, and always in the case of the bodies.

The mixture was allowed to stand 24 hours. A shorter time, such as two hours, and a longer time, as 48 hours, were also tried. The short period was found to be insufficient for the more sticky clays, as these would ball-up more or less in the blunger. The longer period did not seem to help. For the sake of uniformity, all clays, sticky or not sticky, were treated alike.

After the period of soaking, the clays were blunged in a small one-gallon household churn. The amount of blunging depended somewhat upon the clay. All of the mixtures were

blunged at least three or four times for several minutes each time. In the case of the more gummy clays a somewhat more prolonged blunging was necessary. It was found at this point, that the addition of more water was necessary in many cases, as the soaking had caused the clays to take up more water than seemed to be necessary on the first addition. See Fig. 1.

The crude clay-slip was brought up to the consistency of the ordinary slip ready for pumping. This was conveniently determined by feel and by the pouring of the slip, as greater accuracy was not deemed necessary. In the case of several of the crude clays of the more sticky nature, it was found that

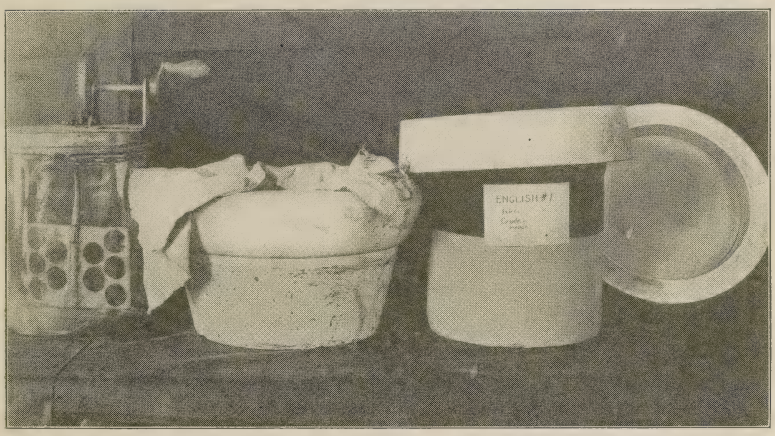


Fig. 1.

even the most persistent blunging did not break up the small clots entirely.

The body mixture was blunged considerably thicker, because of the subsequent screening where additional water was used to facilitate getting the body through.

The body mixture was screened through a 150 mesh screen. (Tyler Standard). After screening, the slip was given one more blunging and was then ready for drying out.

The process of screening brought out one or two rather interesting points. It was rather expected that the sticky, gummy clays, some of which had persisted in holding the small clots

mentioned above, would be hard to get through the screen. As a matter of fact, all of these clays with one or two exceptions went through the lawn quite readily. The slip was placed on an 8-inch screen, about a half pint or less at a time and washed through. The tailings were washed into a separate pan, and finally, when the slip was removed, they were washed again with clear water.

In the case of the majority of the American clays, there would hardly be enough tailings out of the whole batch to justify the second washing. For most part the tailings were flint, spar and grit. But the English clays, and the American most closely resembling them, gave quite a large residue. On account of the ease with which they had slaked down, it had been expected that these would go through the lawn most freely of all. The Special and the Black and the Knowles' No. 12 left something like 200 grams of residue. The English No. 1 came next with about 150, and the Tenn. No. 3 followed with about 60 grams.

Although some residue had been expected because all of these clays contain a considerable amount of what is loosely termed "dirt," it was thought that it should not be so large a proportion as this. It would hardly seem likely that such a large percentage would go over the lawn commercially, and inquiry does not show that such is the case.

Repeated washings get more and more of the residue through the screen. The residue settles to the bottom like so much flint. Twenty to twenty-five grams of flint and spar together would be a high estimate for that part of the residue. In the case of the English No. 1 and the Tenn. No. 3, a goodly portion, on inspection, seemed to be grit and undesirable material. The remainder, however, especially in the case of the Black and Special, appeared to be quite plastic, and carry very little grit.

Going on the theory that the plastic residue was probably caused by coagulation, by reason of organic acids, which cause a differentiation in the size of grain, caustic potash was added. When the residue would be shaken up in clear water the stuff would settle very quickly, leaving the water above quite clear. The addition of the hydroxide caused the residue, or at least

a portion of it, to settle more and more slowly, until a point was reached where the liquor remained black in color indefinitely. Portions of the air dried residues of a couple of these clays were ignited. Black smoke came off for some time, a brown colored deposit collecting on the crucible lid and remaining for a time. The Black residue lost 20 percent of its weight, the English No. 1, 19 percent, and the Tenn. No. 3, 11 percent. All burned to a buff white, and the Tenn. No. 3 showed a tendency to redness.

Since the percentage of clay going over the lawn commercially, can hardly be so high as these screenings indicate, it is presumed that the prolonged blunging in the commercial blungers, breaks up this coagulation to a certain extent, and the use of lawns of coarser mesh takes care of a goodly percentage of the rest. Then, too, if the percentage of this sort of clay used in the body is not high, it probably becomes mixed more thoroughly with other materials.

The bodies mentioned were finally ground for about an hour in small ball mills, after which they screened quite readily, leaving about the usual amount of residue.

After the blunging, both the crude clay and the body mixtures were poured into an old mold of suitable shape, lined with a cambric cloth, so that the clay would not come in direct contact with the plaster. They were allowed to dry out to a state of workable plasticity, checking against the regular factory body as closely as possible by feel, knife cut, etc. In the case of one or two clays, a settling out of the spar and flint was noticeable after this standing. While this method of testing introduces error with a few clays, it should not necessarily militate against the clays, since in practice the clay is kept in a state of agitation up to the filter press, and given no chance for fractional settling. This separation, however, seems to be a rare occurrence, and was overcome as much as possible by thorough mixing while still too soft to be workable.

As the mass comes from the cloth it is likely to be dried out unequally. Hence the next step was to work up the mass rather thoroughly and allow it to age.

The mass was worked into a ball, wrapped in a damp cloth, and dropped into a gallon earthenware crock or jar, over which a water-soaked plaster lid was placed. This latter serves as a miniature damp closet.

The mixtures were allowed to age about three days with an intermediate working, and a final one. The working of the clay, both at this point and later when cutting into briquettes, was done on a glass plate, to eliminate as much as possible the escape of moisture, as would be the case perhaps, with a plaster slab.

From the standpoint of shrinkage measurements, this drying out to reach the workable plasticity stage, and the subsequent working, etc., introduces a large source of errors; in fact, the whole process involves the personal factor to a high degree. Hence, the water content and the shrinkage as a matter of course, is sure to vary considerably.

In order to find out about what this would run, the cuttings from the briquettes were immediately weighed, and then allowed to dry out, and reweighed, and the moisture content figured.

TABLE I—WATER CONTENT

CLAY	DEC. 3		FEB. 2	
	CRUDE	BODY	CRUDE	BODY
	percent	*percent*	*percent*	*percent*
Eng. No. 1	38	21.1	31	19.9–19.3
J. & P. No. 11	29	21.2	30	21.9
Tenn. No. 3	....	21.7	32.6	21.3
Sant. KT.	28.2	20.7	28.9	21.2
Tenn. No. 7	27.4	22.6	27.2	21.3
Sant. F.	27	....	29.1	19.6
Old Mine No. 4	....	....	30.1	20.4
Black	....	....	43.0	26.4
Special	....	11.7*	35.7	26.7
Pink	....	....	33.3	22.9
Cooley	....	....	28.7	21.9
Knowles No. 12	....	....	28.4	20.4
Geo. China.	26.6	....	....	....
Acme Wash.	29.8	....	....	....
Sant. China.	28.6	19.3	....	....

* Screened. Only about half the clay went thru.

In such cases in Table I where the data is comparable, it will be seen, that with one or two exceptions, the same sort of mixture for the same clay, gives the same figures within a few tenths generally, and these were taken two months apart. Also, it will be noted that clays showing more or less similar physical characteristics in other ways, require practically the same amount of water of plasticity. This is especially true of the body mixtures, which give figures more nearly alike for the different clays, the 50 percent of spar and flint tending to equalize the peculiarities of the individual clays.

From these figures, also, it would appear improbable that there would be any very radical differences in the shrinkage

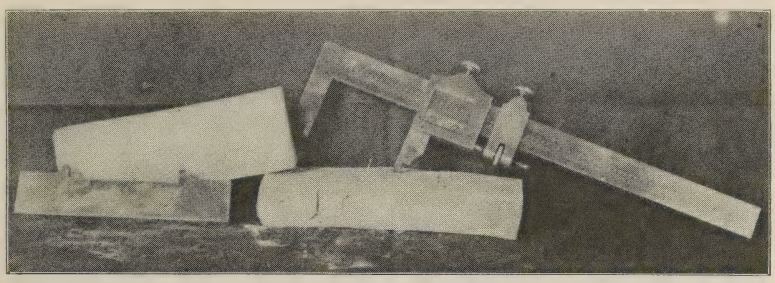


FIG. 2.

readings, and as a matter of fact, as will be seen later, there is not a very great difference.

The mass was now batted out flat and cut with a spatula into briquettes of a size approximately 1 in. by 1½ in. by 5 in. The batches generally yield 6 to 8 briquettes, and 150 to 200 grams for moisture determination. In starting the investigation, very small briquettes were tried, but nearly all of these warped and cracked badly in the drying and burning, and in general, gave such unsatisfactory results that the larger briquette was chosen.

Since the briquettes were not to be subjected to strength tests, it was not thought necessary to use molds in making them. Moreover, as it afterward showed, the extra packing and working required in the use of a mold would most certainly introduce

larger error in the shrinkage determination unless handled very carefully.

The gauge used in making the shrinkage marks gives impressions 5 cm. apart. It is a home-made affair, worked up on a planer in the machine shop, out of a piece of quarter inch scrap steel. The piece of steel was planed down leaving two projections, such that the inner faces are 5 cm. apart. The planer is accurate to within 1/1000 of an inch, giving more than ample accuracy for such a gauge. See Figures 2 and 3.

Immediately after the cutting, the briquettes were transferred from the glass plate to a piece of ordinary oiled stencil paper, and marked with the necessary data and shrinkage marks.

The projections of the gauge make an impression slightly

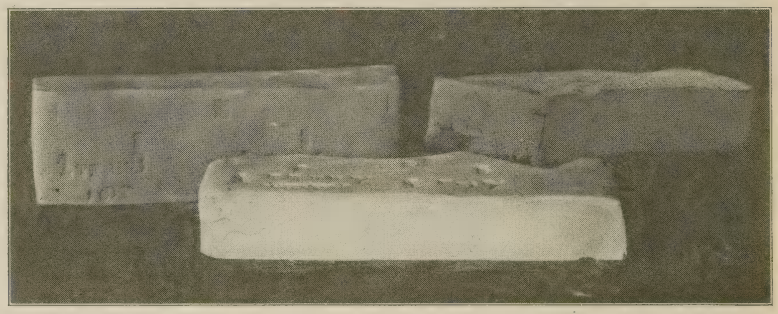


FIG. 3.

larger in area than the caliper jaws. Only the lightest pressure is used to make the marks. Greater pressure bends and pushes the clay around the impressions too much. It was noted at this point that if the mixture was the least bit too soft it tended to stick to the gauge, while if too stiff, it tended to bend. This gives one more check on the point of workable plasticity.

Undoubtedly, calipers with needle points for jaws, and a marking gauge giving corresponding conical holes for impressions, would be still more accurate, but in view of the results obtained with the instruments used, the finer instruments would probably be an unnecessary refinement.

The briquettes were allowed to dry out under ordinary atmospheric conditions for about an hour. This served to stiffen

them up enough to enable them to be turned on edge. If turned sooner they are liable to bend, and if allowed to go too long they will start to curl. These were placed in the commercial driers and allowed to dry as long as is usual for ware of that size.

At this point, the process differs radically from the laboratory method. In the laboratory, these briquettes would be subject to a high temperature for a number of hours and brought to constant weight. Although the varying conditions of relative humidity in the commercial driers introduce error, and do not get rid of as much moisture as in the case of the constant temperature oven, at the same time this is the factory practice and should give figures which may be used at once in the factory. Of course, unusual drier conditions should be avoided.

The briquettes, after the drying, were examined, and the drying data collected.

The first trials had three shrinkage marks on the face. Nearly all of the briquettes showed variation in these three marks. This was especially true of the crude clay mixtures. Sometimes an increase of 1 percent to 2 percent would occur, basing the increase on the lowest figure. This would mean an actual difference of shrinkage of 2 percent to 4 percent. Usually, the differences of shrinkage would be about 1 percent and occasionally there was almost no difference at all, although this was rare in the case of a crude clay mixture.

It was observed in cases where the mass had been batted out a bit thinner, that the readings were sure to vary between the thick and the thin parts, generally as much as a percent or even more. If this same thinness showed on the other briquettes of the same batch then the readings on the thin parts would not differ more than a few tenths of a percent, from those on the corresponding thin parts of the other briquettes and the same was true of the readings on the thick portion.

After this was noticed on the first trials, some of the second trials were also marked on the edge which had been touched by the knife only. These readings all proved to be quite uniform indeed, differing by a few tenths of a percent.

Taking all of the readings of the briquettes together, it was found that the variation of all the percents never got much over 1 percent. Even in the case of batches made up a couple of months apart, the variation does not generally run over 1 percent and in only one case so far observed was as much as 3 percent.

The bodies run much better. Of 16 sets of body trials, 2 had a difference of 1.2 percent, 8 of 1 percent, 3 of ½ percent, 2 of ¼ of 1 percent, and 2 of $^1/_{10}$ of 1 percent. Of 8 similar batches run two months apart, 1 had variaton of 2 percent, 3 of 1 percent, 1 of ¾ percent, 2 of about ½ percent, and one of ¼ percent. These figures are not quite those stated, varying a few hundredths up to a tenth of the actual value.

To be sure that this was not entirely due to the process of working, some of the regular body used in the factory, ready for the cutting for use, was made into briquettes, the only working that it received being to spread it out and cut into briquettes, handling the mass as little as possible. The same sort of variation on each brick and with the other briquettes took place. In fact the average figures on one briquette were a full percent below the figures used to go by in the factory.

It would appear from these figures, that in the process of manufacture, although the general average shrinkage has a certain value, yet the handling in the plastic state, and the varying humidity of the driers, cause a variation of shrinkage, not only from time to time, but on the same surface of the same piece of ware at any given time.

The kilns used in burning were up-draft, gas-fired pottery kilns. The length of burn in the case of the first trials was about 70 hours and in the case of the second trials, 66 hours. The briquettes were divided into three sets, one being placed in the kiln where it would receive a relatively soft burn, the second a medium burn and the third a fairly hard burn. The whole three sets of the first trials received an unusually soft burn, the one set hardly getting cone 7 and the hardest set just about getting cone 10. As cones were not placed in the saggers with the first set the cone temperature was judged by the regular kiln cones nearest.

The second trials received cone temperatures about as follows: first set, cone 8, the second set, cone 9, the third set, cone 10. In this latter case cones were placed in the saggers along with the trials.

Cones should always be placed near enough to the saggers containing the trials to be sure that the cone temperature is known fairly closely. This along with the length of the burn will give a good idea of what may be expected in the way of vitrification of a clay, and allows comparison with tests run in other kilns at other times.

After burning the briquettes, they were once more examined and measured for shrinkage.

As far as the clays so far tested are concerned there was little difference in any of them in the matter of warping and cracking. The color and the vitrification both varied, of course. The vitrification was determined not only by appearance, but also by absorption tests. The briquette was weighed, soaked 48 hours, reweighed, and the percent increase figured. The color has not been compared to any standard, and so far no attempt has been made to classify very sharply.

In the matter of shrinkages the figures on briquettes of the same mixture were averaged and the percent shrinkage figured for each of the three sets. The figures for similar mixtures of all three sets were then averaged and the results given in the table below.

The variation on each briquette was relatively the same as in the case of the drying shrinkages. There was also a variation between the different sets. In the first trials, these latter variations were about as follows: out of twelve batches, one had a difference as great as 3.8 percent, one of 3½ percent, two of 3 percent, three of about 1¾ percent, two of less than 1½ percent, and three of 1 percent. These were all crude clay batches.

Out of nine body batches, two had a difference of 2 percent, four of 1 percent or a little less, and three of slightly less than ¾ percent.

The second trials showed the following variations:

Crude trials — one of 2½ percent, three of 1½ percent, three of 1 percent, three of about ½ percent and two of less

than ½ percent. Body trials, one of 1¾ percent, three of 1 percent, four of ¾ percent, two of ½ percent and one of no variation.

In Table II the column marked "Percent loss on firing", was obtained by weighing one briquette of each mixture at the time the drying shrinkage was taken, and again after firing, and then calculating the percent decrease.

The following steps where error is likely to enter should be noted in following such an investigation:

1. Sampling. That is, in not getting a representative sample.

2. Blunging. Here the main error enters with those clays showing a large residue on the screen. It may be necessary in some cases to grind in the ball mill, in order to get a more representative amount through.

TABLE II — DATA

AVERAGE FIRE — CONE 9	NO. 1			NO. 2		
	CRUDE TRIALS			CRUDE TRIALS		
CLAY	SHRINK. DRY	SHRINK. TOTAL	ABSORB.	SHRINK. DRY	SHRINK. TOTAL	ABSORB.
	percent	*percent*	*percent*	*percent*	*percent*	*percent*
Sant. English No. 1.	10.1	22.73	5.40	7.50	17.95	1.20
Sant. F.	7.01	17.4	3.25	4.06	13.81	4.17
Sant. KT.	6.51	16.01	0.24	5.61	13.98	2.91
Sant. Tenn. No. 3..	8.94	20.02	1.53	7.73	18.04	1.26
Sant. Tenn. No. 7..	5.60	16.60	6.04	4.50	12.84	10.82
Ky. Con. Co. Special	9.20	21.86	9.99	9.53	23.66	9.35
Ky. Con. Co. Black.	10.84	24.66	12.09	7.00	18.99	11.15
Ky. Con. Co. Pink..	.....	.....	.....	6.72	16.23	5.47
Ky. Con. Co. Old Mine No. 4.....	8.00	16.90	4.25	7.002	15.47	5.18
Knowles No. 12.....	.....	.....	.....	6.36	15.97	.....
J. & P. No. 11.....	6.48	19.56	1.26	6.63	15.98	4.36
Cooley Ball	.....	.....	.....	7.05	15.68	5.61

3. The largest chance for error is likely to be in not having the right degree of plasticity, or a uniform texture.

4. Errors come in by uneven packing of the clay in making the briquettes, and in turning the briquette on edge. This error may be as large as you please, or with care, it may be kept quite small.

5. The relative humidity of the drier is likely to cause variation.

6. The degree of the burn and the position in the kiln are the final, and fairly large, factors in the variation.

In the experience of the author, these figures cannot be duplicated unless the method is followed quite closely, and then, of course, the figures will be relative, depending upon factory conditions. Yet, it is believed the process will give valuable aid in the selection and substitution of various clays.

SHEET — BALL CLAY

NO. 1			NO. 2			LOSS BY BURN	
BODY TRIALS			BODY TRIALS				
SHRINK. DRY	SHRINK. TOTAL	ABSORB.	SHRINK. DRY	SHRINK. TOTAL	ABSORB.	CRUDE	BODY
percent	*percent*	*percent*	*percent*	*percent*	*percent*	*percent*	*percent*
5.48	12.81	.020	5.52	11.49	1.23	16.3	6.26
....	.....	....	6.50	12.93	2.52	14.59	7.36
5.11	13.70	0.032	6.15	14.53	0.045	11.82	5.76
6.80	14.71	....	8.01	15.56	0.55	14.51	6.94
5.58	14.60	0.067	4.83	12.75	0.84	11.61	6.34
5.67	14.73	0.065	7.77	18.21	0.056	22.46	13.22
5.94	15.52	.....	6.90	17.58	0.072	12.22	11.91
....	.....	.....	6.17	14.34	0.083	13.37	6.97
5.80	14.45	.....	6.08	14.20	0.033	12.98	6.49
....	.....	.....	7.40	13.77	.....	15.53	.....
5.72	15.79	0.03	5.30	13.40	0.25	13.33	6.89
....	.....	.....	6.33	13.85	0.48	11.94	6.35

A CONTINUOUSLY OPERATED TUNNEL KILN FOR HIGH-GRADE CLAY WARE

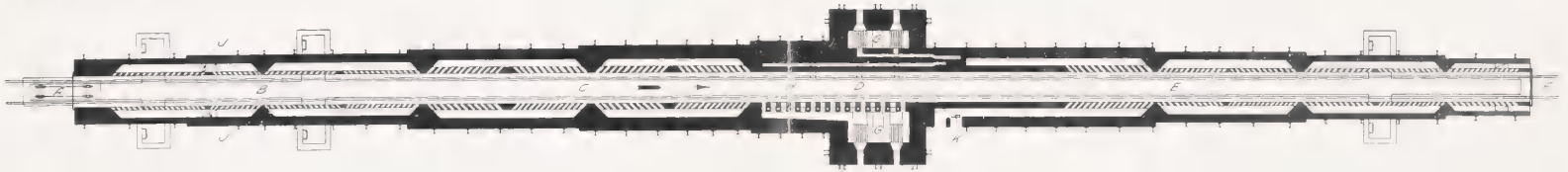
BY LAWRENCE E. BARRINGER

Theoretically, continuously operated kilns have substantial advantages over periodic kilns. In the continuous system the first great advantage is that of fuel economy. There is not the great loss of heat and of fuel attendant upon the heating and cooling of the entire kiln, as in the periodic type. A very considerable percentage of the total heat units obtained from the total fuel used in firing periodic kilns is consumed in heating up the massive masonry construction of the kiln. A second great loss of heat units in the periodic system is the escape of waste heat from the chimney and one has only to observe the "tassel" or flame emerging from an ordinary potters' kiln on a dark night to appreciate the tremendous loss of heat units in this system. These very high losses, due to absorption of heat by the masonry of the kiln and escape of highly heated products of combustion, are shown in Mr. Bleininger's article on The Heat Distribution in four Industrial Kilns in Vol. X *American Ceramic Society Transactions.*

Secondary advantages of a continuous system of firing are lower labor cost of operating, better regulation so as to secure greater uniformity of temperature, lower cost of maintenance and increased life of saggers used for high-grade wares.

Many types of continuous kilns have been proposed in the past, and in some lines of the ceramic industry certain types have been employed with considerable success, particularly throughout Germany, both for low-grade ceramic products, such as brick, and for high-grade wares such as porcelain and stoneware.

Probably the first tunnel kiln was constructed for the firing of over-glaze colors at Vincennes, France, in 1751, as mentioned by Hellot, in his *"Rapport au roi Louis XV"*. The first inventor applying the idea to the manufacture of bricks, etc., was un-



DIAGRAMATICAL PLAN OF KILN
FOR BURNING HIGH GRADE REFRACTORIES

FIG. 1A.

doubtedly Yordt, who took out a Danish patent in 1840. Borrie at a somewhat earlier time, designed such a furnace for hollow ware, taking out the gases through a stack at the middle of the kiln (*Notizblatt 5, 416*). These types were followed by the design of Pechine who located his furnace at one end of the tunnel and then transferred the cars to another tunnel parallel to the first, where they were allowed to cool, (*Wagner's Jahresbericht 1848, p. 269*). Another proposed tunnel kiln was that of Demimuid and Gueval, which embodied the principle substantially as used today.

In 1873, Otto Bock built his first tunnel kiln in Denmark. A German patent was granted Bock in 1877, and he then built about 60 kilns of this type most of which proved failures. The failure of these kilns has been ascribed to the fact that the ware was not properly dried and also the running gear was frequently overheated through insufficient protection from the kiln gases.

In general the various proposed types of continuous kilns may be grouped in two classes, namely:

1. Kilns in which the ware is stationary and the firing zone movable.

2. Those in which the firing zone is fixed, or stationary, and the ware is moved.

In this country a number of continuous kilns are in successful operation for brick, usually of the first-named type. In this work, however, the necessity of uniform temperature is not as great as in the higher grade clay wares, since matters of porosity, exact size and uniform color are not as important. As a result continuous kilns with either chamber or tunnel construction can be operated successfully where the results would not be satisfactory with the higher grade clay wares.

While at Union Furnace, in Ohio, in 1902 the writer witnessed the successful operation of two continuous kilns for dry pressed, buff and grey, face brick. These kilns were of the Guthrie type. A few years previous to that time the writer had also observed the use of a Youngren kiln for ordinary red building brick at Washington, D. C. That the use of the Youngren kiln has grown is indicated in the paper by McElroy and Mumma

in Vol. XVI *Transactions American Ceramic Society.* In both these types the fire zone is movable.

Up to seven or eight years ago there were no continuous

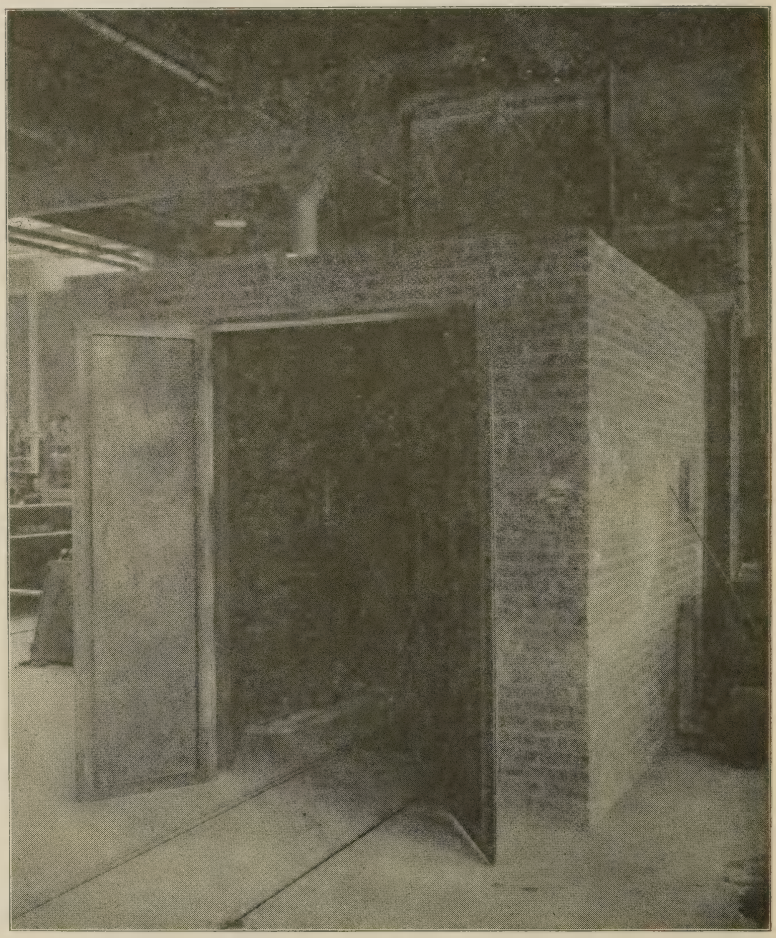


Fig. 2.

kilns being successfully operated in this country for high grade clay wares, as far as I have been able to ascertain, and about that time the writer became interested in the continuous kilns

being used at Altwasser, Germany, for the firing of procelain and stoneware. About the same time an article appeared in the *Clay Worker,* describing these kilns, (*Clay Worker* for April 1908, article by Geijsbeek).

In July 1908 Dr. W. D. Coolidge, of the Research Laboratory of the General Electric Company, who was then in Europe, was asked to examine these kilns at Altwasser and reported there were two kilns in operation at the factory of C. Tielsch and Company, which is said to be the largest all-porcelain (no stoneware) plant in Europe, employing 1400 people and operating 14 periodic kilns and two continuous kilns. One kiln was employed for biscuit firing at a temperature of 1050° C. and the second kiln for glost firing at a temperature of 1430° C. Excellent results were being secured, both as to low fuel consumption and in the matter of maintaining uniform temperature.

At that time similar kilns were in operation at Levano, Italy; Grimstadt, Bavaria, and Montereau, France, for the firing of earthenware, some six or eight in all.

Since that time two kilns of this type have been constructed at the Henningsdorf factory of the Allgemeine Elektricitäts Gesellschaft, in Berlin, for firing electrical porcelain and in this country the first two kilns of this particular type were constructed at Keasbey, N. J., in the works of the Didier-March Company, where the kilns are employed for firing refractory products.

The construction and operation of the two kilns at Keasbey (1910), brought this type of kiln to a point where it could be closely studied under conditions of American fuel, refractory materials, labor, etc., and after a further study of the kiln the writer recommended its construction by the General Electric Company. In 1913 such a kiln was constructed at Schenectady, N. Y., and has been operated with very successful results since that time for firing porcelain.

The origin of the particular details of construction of the type kiln under discussion is somewhat obscure, but certain basic features seem to have been developed in France by E. G. Faugeron of Montereau. Other features have been developed by the Didier-March Co. and their European associates.

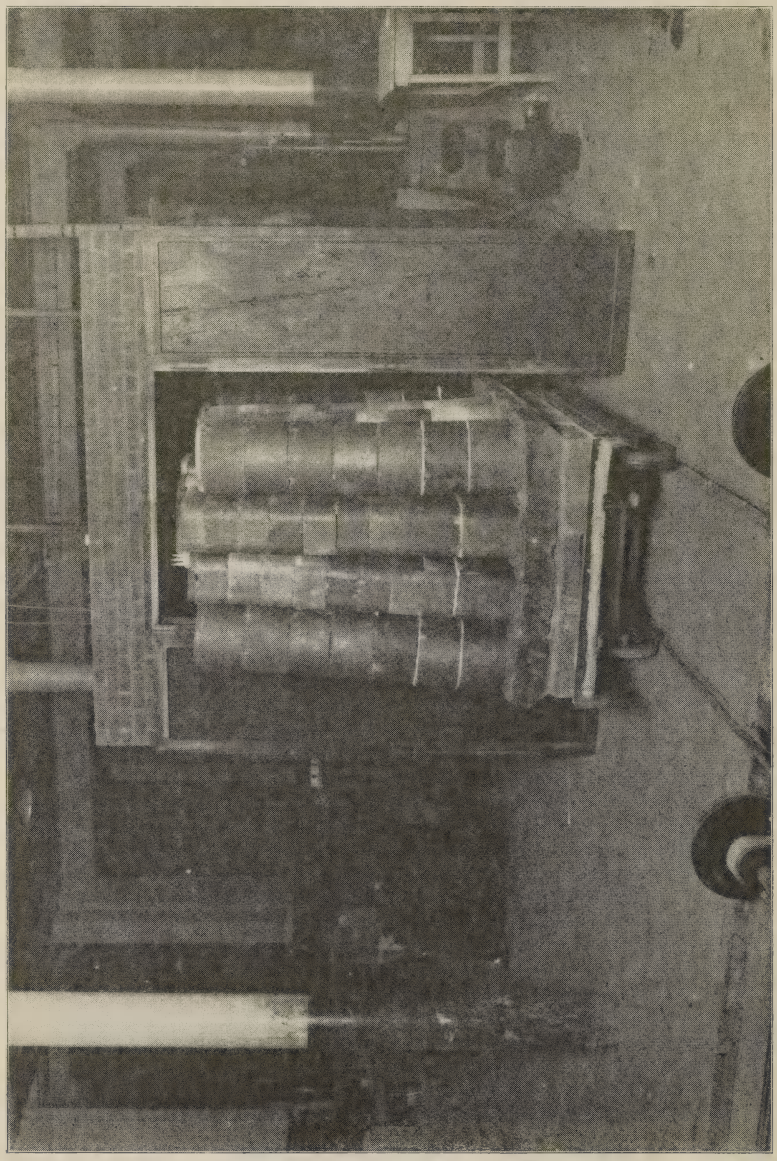


Fig. 3.

While the continuous system of firing is fundamentally sound and of correct principle, application of the system to high-grade clay wares, using high temperatures, has not always been readily accomplished because of mechanical difficulties. There are problems of properly setting and moving the ware, of controlling the draft, etc.

The type of kiln under discussion appealed to the writer because of having seemingly reduced the construction to the basis of the greatest possible simplicity. The kiln is simply a straight tunnel 197 ft. in length with a fixed firing zone somewhat nearer the discharge end than the charging end. A track extends through the kiln and upon this the cars are moved, being gradually brought up to the firing zone, given the necessary period of exposure to the maximum temperature and then passed to the cooling end of the kiln.

By referring to the diagram in Fig. 1, the general operation of the kiln may be explained as follows:

At the charging end of the kiln "A", loaded cars are periodically "fed" into the kiln, being first placed in a vestibule which is shown in Fig. 2. This vestibule is necessary to prevent disturbance of the draft while the inner door of the kiln is opened to receive the loaded cars. After the entering car is in the vestibule and the outer door closed, the inner door is then opened and the car moved into the kiln proper, pushing all preceding cars before it.

Movement of the cars is accomplished by a hand windlass operating an endless chain just below the cars at the charging end of the kiln. This chain is provided with hook lugs which engage the axles of the car and move it forward as the windlass is operated. To facilitate the pushing of the entire train of cars the track through the kiln floor has a slight drop from the charging to the discharging end.

As each additional car of ware is placed in the kiln the entire "train" of cars in the kiln progresses car-length by car-length, gradually passing through the zone B, which for some wares might be called the "water smoking" zone, into the zone C and thence into the firing zone D where the highest heat is imparted to the material. After the car of ware has remained

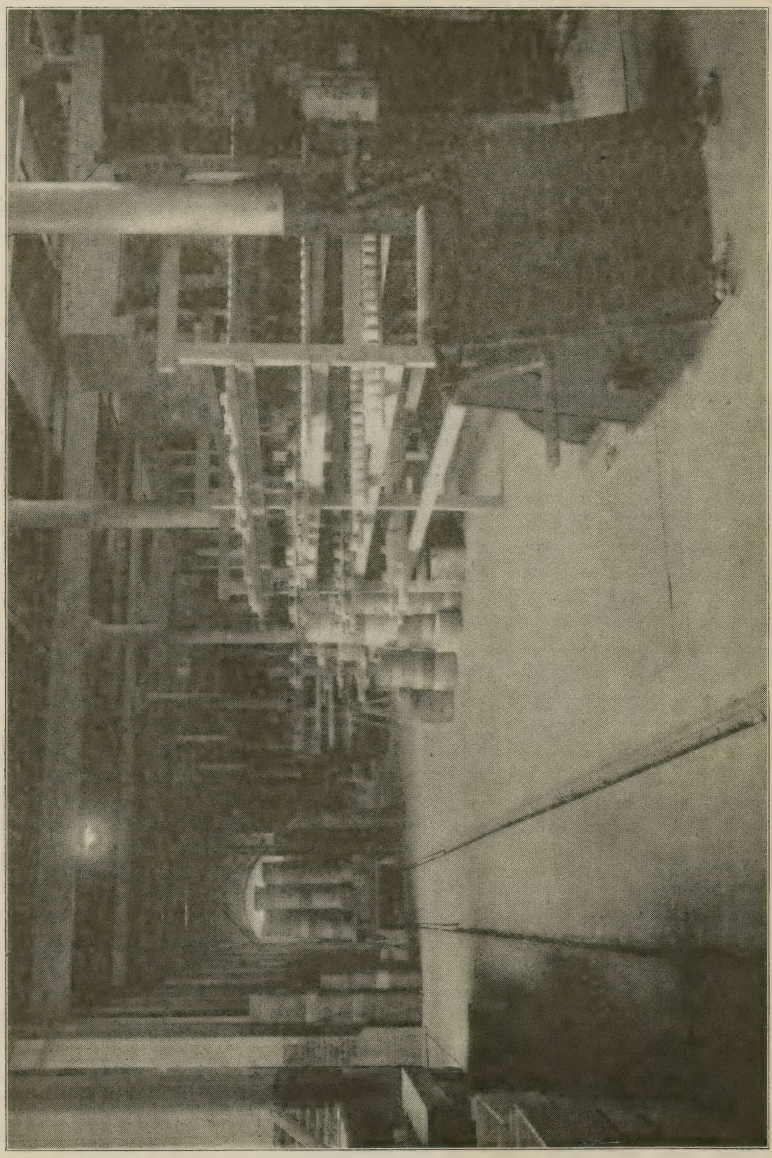


FIG. 4.

in the firing zone a sufficient length of time to bring about the desired vitrification or shrinkage in the ware, as indicated by pyrometric cones, or other means, it passes into the cooling zone E where the material is cooled slowly and uniformly. The car leaves the kiln at F in such condition that the material can at once be taken to storage and unloaded, or, if somewhat too hot, can at least be sidetracked and gotten out of the way of the kiln, thus providing an advantage in this respect, since in periodic kilns too hot to unload, the kiln space must be occupied until the men can take out the ware.

At a point "G" between the charging and discharging ends of the kiln the four grates for direct firing are located, two on either side, and form the base of the combustion chamber. This combustion chamber is separated from the main tunnel by an inner wall, and the products of combustion pass through the tuyeres H and enter the main tunnel where they become diffused and circulate around the ware, successively passing through and into the zones C, D and B. At the point J the waste products of combustion and steam pass out of the kiln into the stack.

Air for combustion enters the kiln through ports in the walls at the discharge end. This air passes through "serpentine" flues and becomes preheated by contact with the cooling ware. As the ware cools, the heat units are given up to the in-coming cold air, and this preheated air passes on until when nearly to the firing zone it is divided, part of the air passing under the grate bars to be used for combustion (primary air) and part of the air being deflected into the kiln chamber to complete combustion of the fuel gases (secondary air). The heated combustion gases pass through the tunnel towards the charging end and heat up the in-coming ware, leaving the kiln through the stack flue at a point about 10 feet from the charging end of the kiln, as indicated by J in Fig. 1.

The kiln has a capacity of 36 cars, and one car is entered and one removed from the kiln every two hours. A car thus requires 72 hours to pass through the kiln.

An important feature of the cars is the "sand seal" to prevent the heat of the kiln from finding its way beneath the cars where it would soon damage the wheels and axles, to say

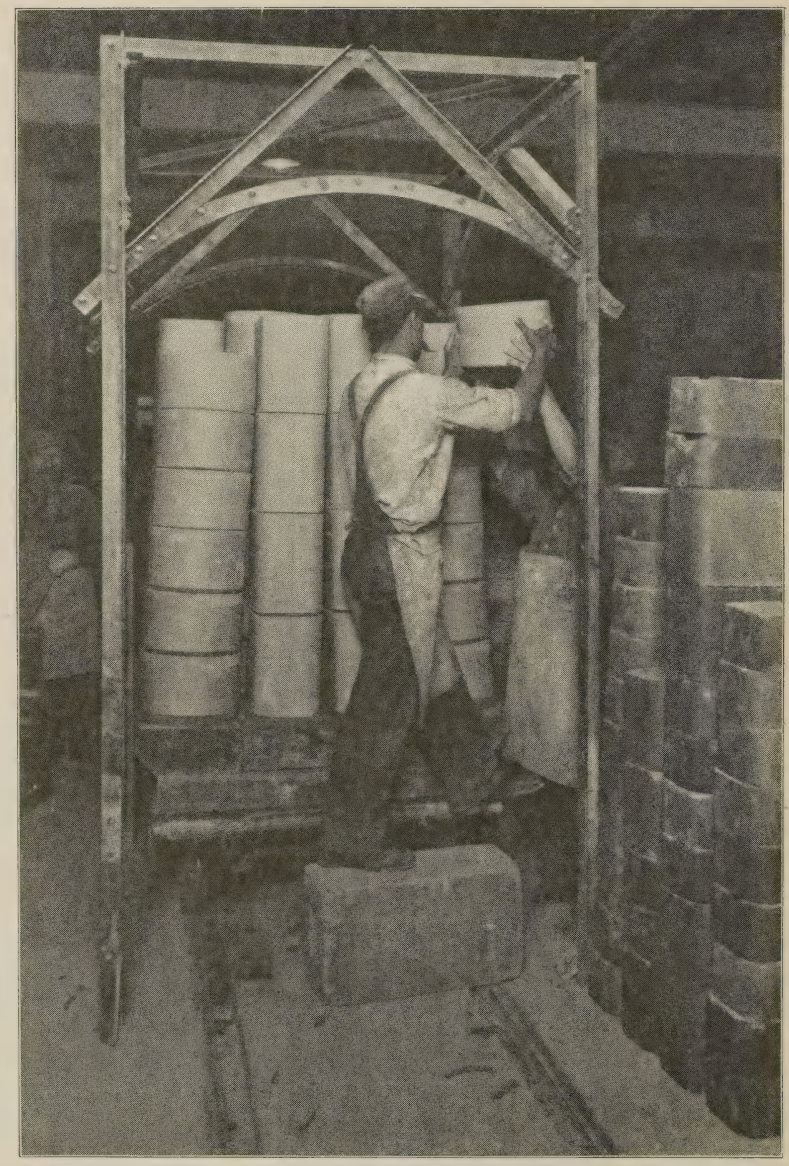


FIG. 5.

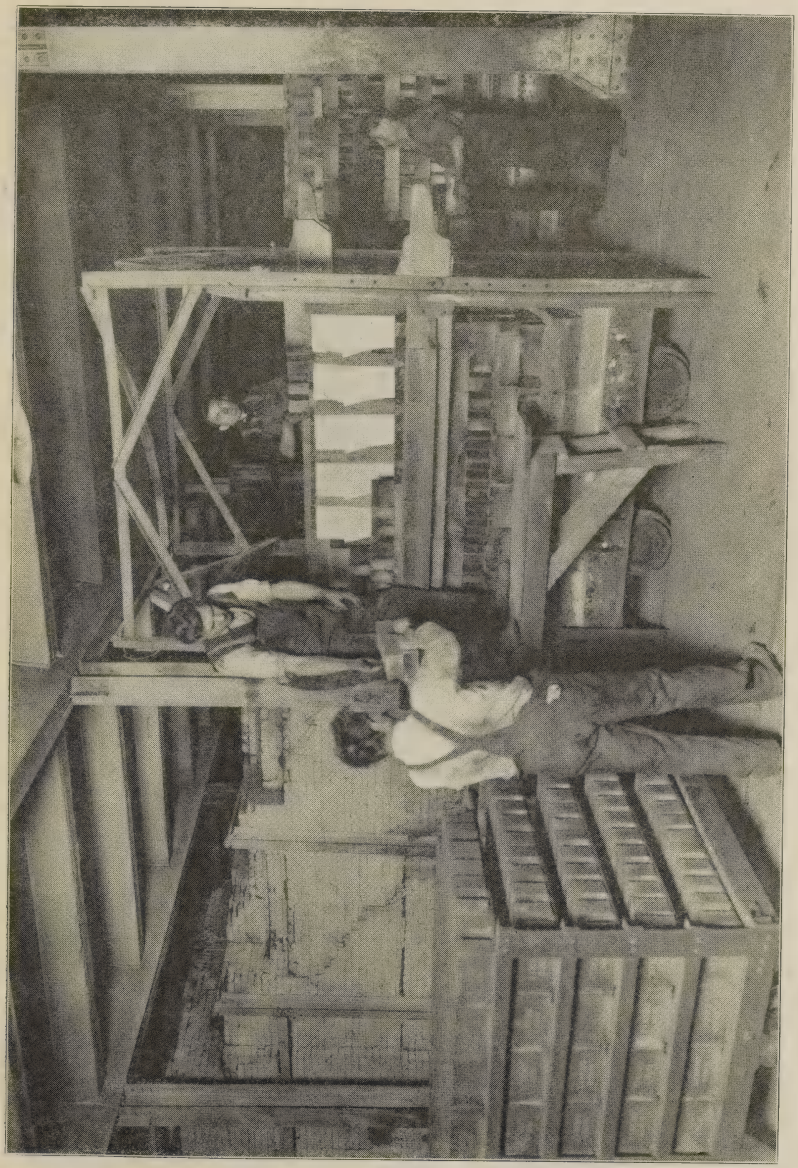


Fig. 6.

nothing of disturbing the draft. This consists of sheet-iron aprons or shields which move through troughs of sand on either side of the tunnel wall.

The tunnel is provided with a "subway" which extends the entire length of the kiln beneath the cars and from which the heat is excluded by the sand-seal above mentioned.

The temperature is measured by a Taylor Instrument Co's recording radiation pyrometer and also pyrometric cones placed on each car. The recording radiation pyrometer is used to maintain the correct firing conditions to give the greatest possible uniformity of temperature, and the seger cones are used on each car as a final gauge to determine when the car has been in the firing zone for a sufficient length of time. While the normal time for each car is 2 hours, it is sometimes necessary to remove a car from the firing zone before this period elapses, and at other times a longer interval is required. These differences in firing periods are brought about by differences in fuel, different draft conditions, bulk of ware being fired, etc., but in general the periods average two hours.

The draft is indicated by a draft gauge placed at the entrance of the stack flue. Draft in the kiln is controlled by dampers, both for the primary and secondary air, and it can also of course be controlled at the ports through which the cold air is admitted to the kiln at the discharging end. There is also a main damper controlling the draft in the flue to the stack or chimney.

Youghiogheny coal (1¼ in.) is used for firing, and the four fireboxes (two on either side of the firing zone) are fired one at a time every 12 to 15 minutes so as to maintain as closely as possible the same conditions of oxidation and reduction, as well as temperature. About three tons of coal are used daily.

Figure 2 shows the vestibule at the charging end of the furnace.

Figure 3 shows a car entering the kiln.

Figure 4 shows the hand windlass by which the entering car is pushed into the kiln and also, in the background, shows the loading gauge and the racks from which the saggers are filled with ware before they are placed on the cars.

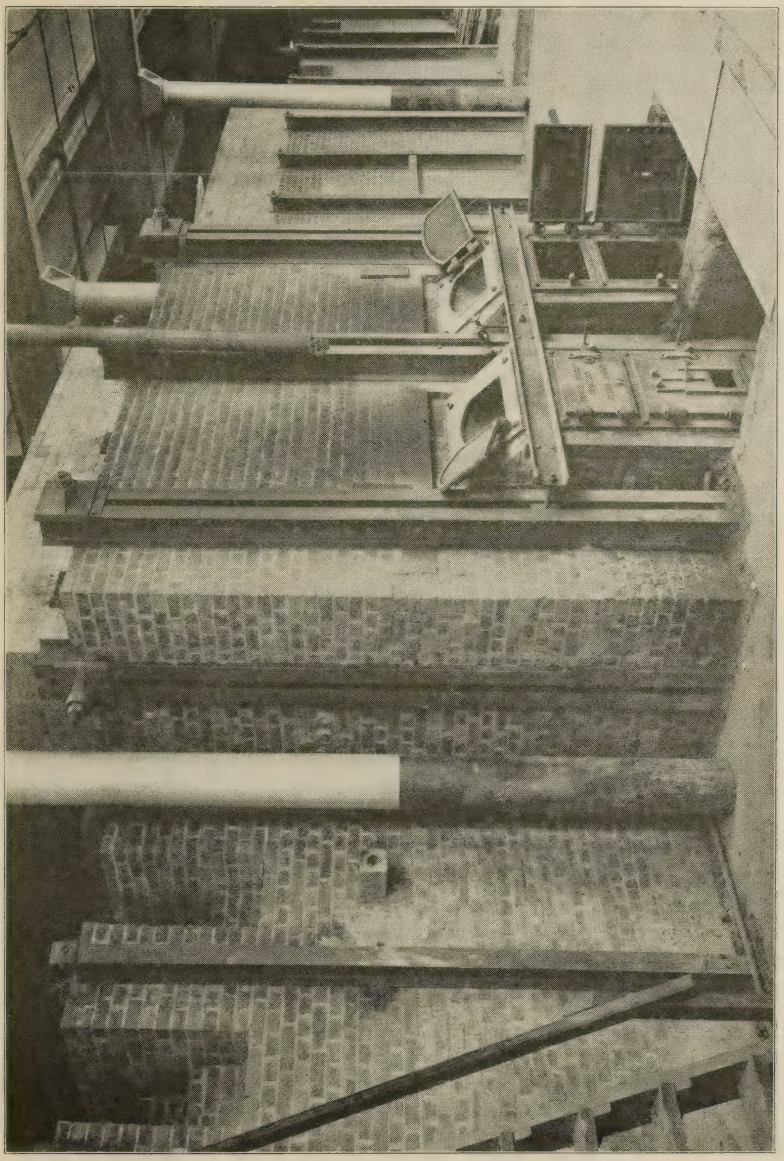


FIG. 7.

Figures 5 and 6 give close views of the loading gauge used in one instance for loading the cars with saggers of electrical porcelain and in the second instance with fire brick and refractories.

Figure 7 shows the fire-box zone.

Figure 8 gives a general view of the kiln from the discharge end, and Figure 9 of a car emerging from the discharge end of the kiln.

Figure 10 shows a car of refractories just withdrawn from the kiln.

The kiln at Schenectady is used in firing of porcelain insulators to a temperature of 1300 to 1400° C., this class of ware of course being all fired in saggers. The kilns at Keasbey are used in the firing of fire brick and refractories at practically the same temperature, and in both instances a uniformity of distribution of temperature is secured of well within one cone.

As an indication of the efficiency of this type of kiln it may be noted that the temperature of the escaping gases is very low, averaging 200-220° C. at the damper in the Schenectady kiln and 150-200° C. at the damper in the Keasbey kilns.

The tunnel and track must be constructed with great accuracy, and it is also imperative that in passing the loaded cars through the kiln there be no obstruction or contact of the cars with the inner walls of the tunnel. In loading the cars the gauges previously described are used constantly, so that the loading can be carried to a maximum without having the ware project at any point so as to strike the tunnel walls.

In the kilns at Keasbey 650 pounds of coal is used to 1000 standard 9 in. brick. In the kiln at Schenectady three tons of coal are used each 24 hours or 21 tons per week. Each car will carry eighty-seven (87) saggers twelve inches in diameter by six inches high (inside measurements). Thus in one week about 7000 such saggers can be handled. The continuous kiln turns out about three and one-half times the work of one periodic kiln of the size mentioned, or, in other words, is equivalent to three and one-half 14 foot periodic kilns.

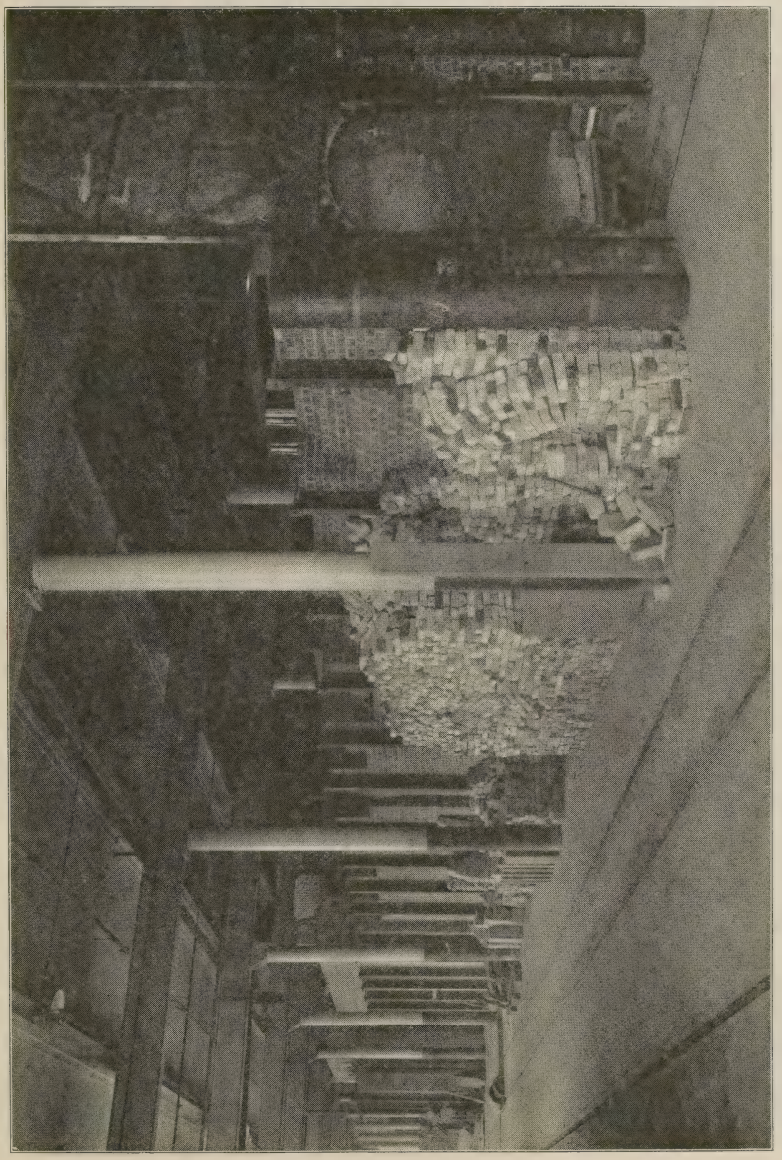


FIG. 8.

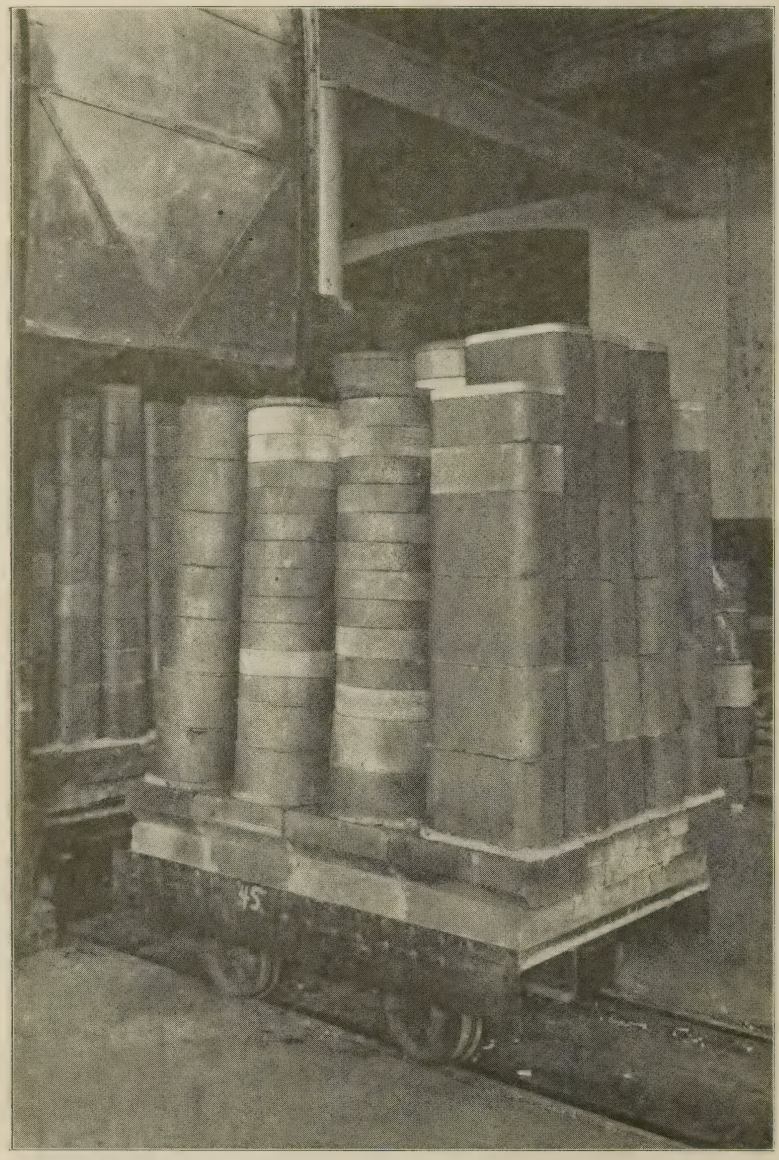


FIG. 9.

The amount of coal consumed is about one-half that of the periodic kilns for the same sagger volume carried through. In computing the cost of porcelain firing, the volume or space occupied is the basis used for calculation.

For those who figure weights it may be said that a sagger of the size under discussion weighs about 31 pounds and the weight of ware will average about one-third of this, giving a total average weight per loaded sagger of about 41 pounds, or a total weight per car of 3567 pounds.

In the matter of labor the cost has been found to be about 10 percent less for loading and unloading, and the cost of firing about the same as in periodic kilns. There is, of course, a greater degree of cleanliness in having the firing zone at a fixed point and of comparatively small area and the loading and unloading can be carried on with greater ease than in periodic kilns.

The life of the saggers has been increased $16\,^2/_3$ percent, due probably to the fact that they are not subjected to the weight and cross strains encountered in the periodic kilns.

As far as our experience has gone with the kiln at Schenectady, the cost of repairs amounts to approximately the same as in three or four 14 foot periodic kilns. Special refractory shapes are required for construction and maintenance, and these are, of course, more expensive than standard fire brick.

The Didier-March Company were the pioneers in building this type of kiln in this country and in addition to constructing two kilns of their own, constructed the kiln now being operated at the Schenectady Works of the General Electric Co.

United States Patent No. 1,103,184 applies to this type of kiln, and it is also covered by a number of foreign patents, among which may be mentioned the following:

German Patent No. 104,241 and 119,516
Hungarian Patent No. 13,504
French Patent No. 275,994
English Patent No. 13,386
Austrian Patent No. 48,365
Swiss Patent No. 17,570

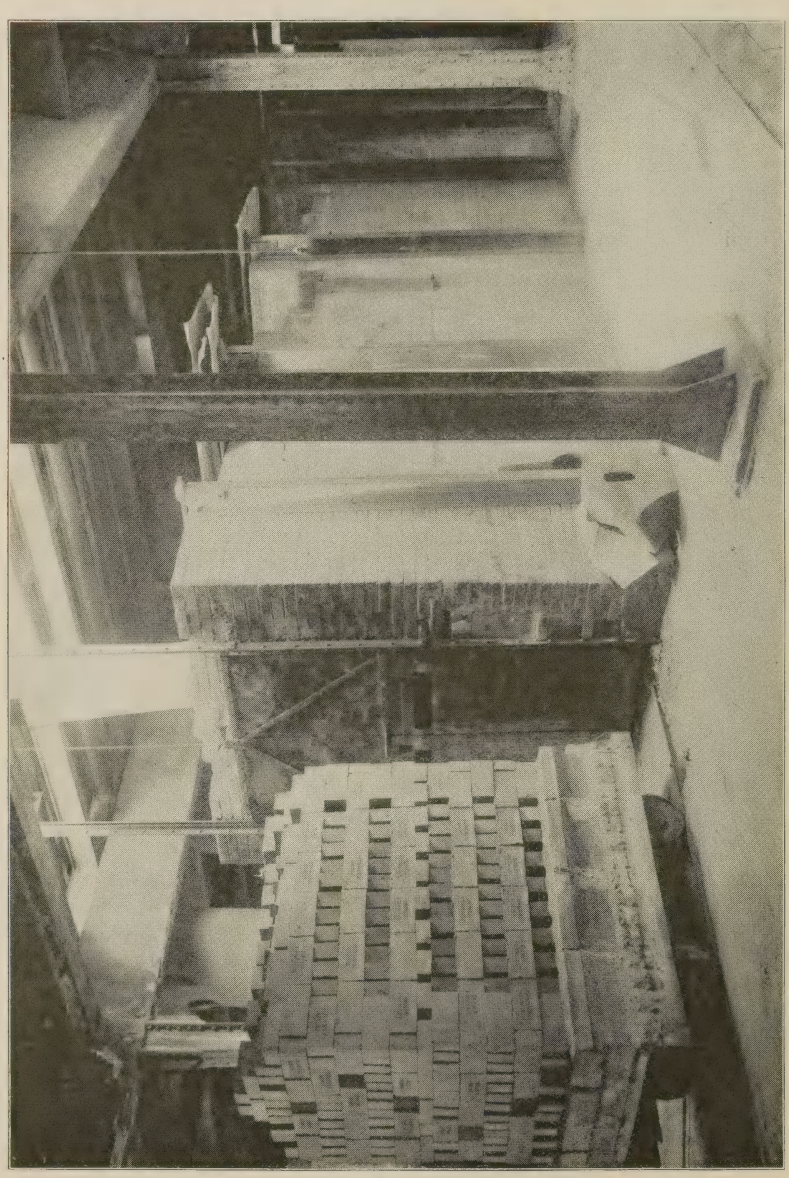


FIG. 10.

Luxemburg Patent No. 3,324
Belgium Patent No. 137,906
Italian Patent No. 106,243 and 162,216

In conclusion the writer wishes to express thanks to the Didier-March Co. for data and photographs, to A. V. Bleininger for historical data and to Prof. Edward Orton for suggestions as to desirable data.

NOTES ON THE ELUSIVE TWENTY PERCENT PAVING BLOCK

BY MARION W. BLAIR, M. E.

There is a steadily increasing demand for paving block of high quality, low test and uniformity. Many plants are so favorably situated as to raw material that a low test, say around twenty, is the rule rather than the exception. Some plants never hope to make a twenty percent block, and by their owners the rattler is regarded as a piece of inhuman ingenuity, designed solely to ruin their business. Some others have been able to improve their quality by care and diligence. There is a third class and the most discouraging of the lot, where, among an average which is high, an occasional eighteen or twenty percent test turns up and the question naturally arises, if one test can be made like that why cannot all of them be made that way.

The importance of this variation is fully recognized by the manufacturer upon whom it may inflict a serious loss. Through fear of such conditions, the manufacturer puts on a price which loses the work, or, having accepted a contract, he loses the profit of a season by rejection and condemnation of certain shipments which he may have shipped in good faith as coming under the requirements. Even under the latest adopted rules of test, resorting may become necessary, and the result will be a loss of profit even though the job is retained. The condition of elusiveness, however, has been accepted as one of the unavoidable curses of the business rather than being subject to control. It has been in an attempt to spot the defect in method or material on such plants that the writer has made a number of experiments and, while they have not been conclusive in their results, they have been interesting and, I may say, profitable. An account of them may prove of interest to others — or give another some idea from which he may locate a baffling defect in his own product. With this in view, rather

than to produce a scientific article on the subject, this paper has been written.

Common sense and a little observation will demonstrate pretty clearly that there is not an atom of uniformity in a thirty foot shale bank. There is shale which has been partially weathered, and there is unweathered shale. There is sandstone in ledges and gravel. There is common yellow clay and grass roots, iron ore, limestone and many other things in the mixture, each and all having its own chemical composition and physical form, and out of the mixture it is impossible by our present methods to extract ton lots to be ground, screened and pugged, run into a column, cut into ten pound sections, pressed, dried, burned and shipped, from which any ten of the ten pound sections may be picked which will in all particulars be exactly similar to any other ten sections which have come from the same pit but probably from a different ton. Paving brick manufacturers know this, sensible engineers ought to know it, and while the *A. S. T. M.* recognizes it in the report of Committee C-3 on Paving Block Tests adopted this year, still the demand continues for an unreasonable uniformity in low testing paving block. The manufacturer continues to try to produce it — not as an essential to quality in the pavement, for that he already has in the brick far in excess of the quality and care used in construction, but as a protection to profits which are lost by condemnation of shipments which are at the mercy of an unreasonable engineer.

In general a clay mixture can be obtained at the dry pans which will produce a column of fairly even color and that is the basis upon which the average plant, and especially a failing one, will lay its claim to an effort to make a uniform product. But a uniform color does not indicate a uniform grain or uniform pugging, or machine knives and auger in good condition, or a properly lubricating die, any one of which "gone wrong" can be cause sufficient for a wide variation of test.

The writer appreciates the fact that no two materials can be worked exactly alike. But it must be conceded that each material has a right way and a wrong way to be worked, each detail having a measurable quality essential to the best in that

particular material. After it has been demonstrated, for instance, that fifty percent or more of a given clay must pass through a sixteen mesh screen to produce a twenty percent block even part of the time, there should then never be found on that plant a block of coarser grind, except its manufacture be the result of accident or experiment, and in either case a record of when and why should be possible. That is the ideal, but unfortunately the most approved grinding and screening apparatus now in use is not capable of such fine adjustment, and the present and prospective price of paving block perhaps would not justify either the investment in machinery or the cost in labor to secure it. It is doubtless at the screens that a sensitive clay receives a variation of grain which might easily account for all the lack of uniformity in its testing qualities, and a little care at this point might easily make a fairly good block in materials which are now considered worthless for the purpose.

If these experiments and suggestions point to a greater refinement in screening, mixing and firing methods than the manufacturer is prepared to accept as necessary, they will meet with scarcely less hostile reception and criticism than the theory advanced by the writer in 1910, that a satisfactory and uniform testing apparatus would require a chemical analysis of the shot employed. Today, however, that refinement is above criticism of engineers and manufacturers alike.

The condition which produces the variation is sometimes momentarily beyond control — for instance, wet and dry condition of clay and uneven feed, a loose or a broken screen wire, the grinding of dryer waste. The tests in Table I, taken at one minute intervals for ten minutes when none of the unusual conditions mentioned prevailed, will show somewhat the extent of this variation.

TABLE I — SIZING TEST

	THRU 20	ON 20	ON 16	ON 12	ON 10	ON 8
	512	107	162	100	099	017
	465	086	232	100	096	015
	481	97	160	121	119	019
	480	109	185	115	93	17
	498	92	195	104	93	17
	464	104	249	101	72	08
	612	119	141	73	47	13
	449	96	179	141	119	013
	543	105	171	102	70	007
	554	112	172	091	064	005
Average	505	102	184	104	87	13
Maximum percent...	61	11	24	14	11	19
Minimum percent....	44	8	14	7	4	5

Yet in many plants striving to produce the twenty percent block, there is no method employed to check the screening, and such a thing as a set of chemists' screens is unknown.

The block produced in that ten minutes were marked, set together in a kiln and burned under oxidizing conditions, with the results shown in group I, Table II.

TABLE II — RATTLER LOSSES

GROUP I OXIDIZING BURN	GROUP II REDUCING BURN
25.25	
24.73	
26.80	18.75
22.65	20.05
23.68	18.22
24.62 Average	19.01 Average
26.80 Maximum	20.05 Maximum
22.65 Minimum	18.22 Minimum

A portion of the marked block were then reburned under reducing conditions, with the results shown in group II, Table II.

There is no evidence that the lack of uniformity in group I was entirely due to a lack of it in the grain of the clay but, on

the other hand, there is no reason to suppose that it did not influence it.

Sad experience on one plant has demonstrated that coarse grinding is dangerous and, while it is impossible to secure uniform fineness, it is possible to prevent undue coarseness. Therefore, tests are made four times a day, and should an appreciable increase in coarse clay appear, it is investigated and the defect remedied, be it from a wire loose or out, or what not. It has been found that the wearing of wires allows a gradual increase in size of grain, but this increase is not allowed to go beyond certain limits. Chutes have also been provided which reduce this wear to a minimum. Variation, however, continues as the following tests demonstrate.

TABLE III — SIZING TEST[1]

THRU 20	ON 20	ON 16	ON 12	ON 10	ON 8
492	149	209	92	47	5
531	121	239	70	33	3
607	122	174	69	24	1
632	146	178	35	8	1
486	144	235	100	32	1
476	127	219	116	56	1
647	119	231	39	11	½
671	119	133	48	27	2
614	126	170	64	23	1
568	143	185	76	26	1
628	151	181	31	8	1
576	124	210	71	18	1
359	97	199	133	154	55
529	185	203	57	23	3
538	120	229	69	37	5
639	120	147	55	36	4
558	153	240	33	12	2
575	192	167	43	18	3
533	155	195	75	39	2
518	139	172	98	65	8

[1] Taken at two and one-half hour intervals.

But a kiln with wide variation is becoming a rarer occurrence. With the screening under close observation, attention was turned to other processes in the manufacture.

Pugging is more or less variable, and its variations are more difficult to observe and control. Worn knives, wet or dry

clay, more or less water and variation in grain are all factors. The influence of an even pug on quality, however, is beyond question. But in this particular, as in no other except burning temperatures, is each material a problem unto itself. I have been unable to devise any practical method of measuring the pugging which can be quickly and accurately checked. There is too much deflection in the cutting wires, permitting an uneven quantity of clay in separate blocks, to determine the pug by weight. The following experiments on pugging indicate the influence that different pugging may have. To determine whether the oily waste going back to the pug mill was detrimental or not, I had all the waste during a given period removed from the belt, letting fresh clay only go into the blocks made. Each block was marked.

The waste alone was then returned to the pug mill with no new clay and run into block. Each block was marked in this case as well. I expected an exaggerated difference in test, as the oily condition was exaggerated. The clay was of course identical, as the waste had been part of the original column. The two lots were dried, one block of each lot set faced with one of the other, burned as usual and tested with the results as shown in Table IV.

TABLE IV

SINGLE PUG	DOUBLE PUG
33.6	23.
30.8	25.6
34.5	27.05
33.4	26.
33.84	26.
32.73	25.6
30.00	25.
33.57	26.
32.	25.25
	26.28
32.934 Average	25.578 Average
34.9 Maximum	27.05 Maximum
30.00 Minimum	23.00 Minimum

The lot had been divided and a portion set in a different part of the kiln and as the result was so different from what

had been expected, a second run of tests was made. The results are shown in Table V.

TABLE V

SINGLE PUG		DOUBLE PUG	
35.4		25.48	
33.5		29.2	
33.6		24.2	
38.77		26.3	
32.72		24.0	
28.82		26.0	
31.79		23.43	
33.84		22.95	
31.96		23.90	
34.35		22.36	
33.475	Average	24.782	Average
35.4	Maximum	29.2	Maximum
31.79	Minimum	22.36	Minimum

The only conclusion which I could consistently draw was that the second pugging had overcome any bad effect the oil might have had, but it showed that an unusually low test might easily come from a batch of waste clay being run through the machine. The clay used in this series was a particularly sandy shale which ordinarily gave the higher tests.

Priding myself upon a brilliant discovery, a year later I tried the same thing on another plant, and even ran a portion of the batch through the pug mill a third time. The result follows:

TABLE VI

	SINGLE	DOUBLE	TRIPLE
	21.54	24.33	25.92
	21.27	22.22	25.79
	21.92	23.93	24.73
	21.65	23.52	27.00
	21.16	21.92	24.86
Average	21.50	23.18	25.66
Maximum	21.92	24.33	27.00
Minimum	21.16	21.92	24.73

Comment is perhaps superfluous, except that the opinion that each material has its own best treatment so far as pugging is concerned was materially strengthened. But further investigation on that particular plant in that particular department is certainly in order, for it is known that eighteen percent block have been made there and it is not unreasonable to believe it can be done again.

A paper of this character would not be complete without the mention of our old friend lamination. In this connection, die friction probably introduces an additional variable which is shown in dry slips just under the surface of the block, which are readily exposed in the rattler and an undue proportion of such block in any one test will run that test exceptionally high. On the other hand, their absence will produce a test exceptionally low. This defect is readily recognized and can probably be largely eliminated by a feeder of the disc type over the pug mill, giving an even flow of clay for any set lubrication.

Even though every other department were under observation and working at its best, it is entirely possible to upset all precaution in the burning and cooling. That a different finish — reducing or oxidizing — will produce different results in certain materials has been clearly demonstrated. In this connection I will refer again to the tests in Table II, which show a difference of four points between the two methods of finishing.

The degree of reduction may also show an influence of several points as shown in the following tests:

	(Reburned)
23.03	20.83
25.52	20.72
25.78	
28.57	
27.22	
24.47	

The first two tests and the two reburned were taken from the same kiln zone and adjacent. The remaining four tests in the first column were block from different points of the same kiln.

The temperatures in the two cases were as nearly identical as the use of pyrometric cones enables one to obtain. The only apparent difference was in the length of the reduction period. It is my opinion, though not yet fully demonstrated, that some materials are more sensitive to very slight temperature differences than is supposed and that a careful study would reveal a critical time and temperature at which very nearly identical results could be expected and which is possible to secure commercially with proper apparatus. The difference in test between the top and bottom of kilns indicates this to a certain degree, although the superimposed weight on the lower courses is no doubt a factor.

Many questions arise which can only be answered by the average results of many tests under as nearly identical conditions as is possible to maintain. A systematic record of results so obtained on any particular plant would eventually offer a solution of its problems, but it is a slow, tedious process. The meager results here given cover about four years' study and observation, and we are just beginning to learn some of the points which need to be studied. Therefore this paper can have no proper conclusion. It is a progress report with most of the progress left out. One might be compared to a kitten chasing its own tail, for the block we test today were made thirty days ago. What the block we make today will test will be an unsolved problem for thirty days to come and from now to then we have constantly changing conditions. A block once made is past correction. It is what it is and must so stay. Unlike many iron or steel products which can be tested and corrected either *en masse* prior to pouring, or later by individual heat treatment, paving block can be added to or taken from only to a very small degree in the kiln. Success must depend on a system of records by which today's product may be judged by that of at least thirty days ago, and in turn that of thirty days hence by today's. If a plant has no such system and its organization lacks the ingenuity to devise and ability to interpret one adapted to its need, it is probable that the twenty percent block will continue to be as elusive as a will o' the wisp.

SOME USES OF CERAMIC MATERIALS IN THE ELECTRICAL INDUSTRY

BY W. ANGUS DENMEAD

It was suggested to the writer some time ago that ceramic engineers and producers of ceramic materials, had as a rule no idea of the extent to which ceramic processes, products, and common materials are used in the electrical industry, and that a brief paper on this subject might be of considerable interest to producers of ceramic materials and wares. For your consideration, the following was prepared in which I have attempted to give the major uses as headings with brief ideas of the materials and processes wherever possible.

This paper includes all the uses with which the writer is familiar but there are doubtless many other materials and products in use.

Under the first grouping is given the true ceramic products, in other words the products that depend upon the formation of silicates for their properties to a considerable extent at least. In the second group is given the materials or processes in which the comon ceramic materials, such as flint, clay, feldspar, whiting, etc., are used to a greater or less extent but in which silicate formation plays either a minor part, or none at all. It may perhaps seem to be going out of the field to include the second group, but as this group should be of interest to producers of raw materials for ceramic purposes, it is included.

TRUE CERAMIC PRODUCTS

Electrical Porcelain. This product is probably the most important single ceramic product in the industry as practically all line insulation, as well as a multitude of wiring supplies, such as porcelain tubes, cleats, knobs, switch bases, socket covers, etc., are made of this material. It would appear as if this field

will be developed far beyond its present status, as the strides taken in the past ten years have been very great. This product is almost invariably one-fire work, the glaze and body being matured together. The glaze is similar in formula to the body, but so designed as to melt several cones earlier, thus passing to a glaze by the time the body nears complete vitrification. The choice of mixture and ingredients as well as the preparation is highly important, as it is extremely desirable to reach as near absolute vitrification as possible without deformation. Electrical porcelain possesses certain advantages that will make it very difficult to replace, a few of which are cheapness, ease of forming to many shapes, permanence, heat and water resistance, high dielectric strength and appearance.

Compounds for Heating Devices. The growth of the heating device branch of the electrical industry has created a demand for a material that has a much higher dielectric strength and resistance than porcelain at elevated temperatures. A number of compounds are being produced for this purpose in which an attempt has been made to take advantage of the well-known properties of alumina, magnesium oxide, lava and several other materials for the purpose, but in practically all cases the compound depends upon the formation of silicates during burning for the binder. There is a constantly increasing demand for these materials, and the ceramist is hard pressed to keep up with the electrical engineer's requirements.

Compounds Resistant to Breakage from Sudden Temperature Changes. As a great deal of electrical machinery has a tendency to heat up rapidly, and much of it is placed where it is exposed to the elements, there has arisen a demand for material capable of resisting sudden fluctuations of temperature better than porcelain. In this class are included heating device plugs and element supports, medium temperature electrical furnace linings, as well as spark plugs and the like, the nature of which makes such a material essential. Here again the formation of silicates is depended upon for binding, and materials such as alumina, steatite, zirconia, etc., are added in an effort to impart their characteristics to the whole. This group somewhat overlaps that of the group above, and in general, materials fulfilling

the requirements of one group will answer those of the other, but in a number of instances the requirements are quite different.

Molded Compounds. Lest this term be misunderstood, it may be well to state that by it the writer means practically any material, (excepting porcelain and closely allied materials), which are formed in a plastic, or semiplastic state and afterwards hardened and given the other required properties by heat, steam-pressure, treatment with chemicals, etc.

There is only one compound with which the writer is familiar that depends wholly upon silicate formation for its bond, but this is an extremely useful one, depending upon the sand-lime brick reaction for its success, in that this reaction causes the hardening of the compound and produces low porosity. Fillers such as asbestos may be used in the same to increase its strength, and the compound may be finally inpregnated if porosity must be absolutely eliminated.

Refractory Materials. These are not directly related to the industry, but the development of the electric furnace for various purposes has created a demand for refractories of extremely high deformation point, good resistance to temperature changes and high electrical resistance at elevated temperatures. Among these materials are brick, linings for high temperature apparatus and other refractories, depending upon alundum, carborundum, green oxide of chromium, magnesia, zirconia and many other materials for their refractory properties, but in most cases silicates are depended upon for the binder. There is a limited use in this industry for graphite blocks and tubes, through which large currents are passed, causing rise in temperature. These blocks are used in furnaces where extremely high temperatures are desired, and usually some neutral gas is used for an atmosphere in order to cut down the oxidation of the graphite units, thus prolonging their life. The binder for these blocks and tubes is usually a ceramic mixture. Arc lamp carbons, though not belonging strictly to this class are included here, and though their production is a decreasing one, owing to the series incandescent lighting, it is still great. A ceramic mixture is usually loaded with a conducting substance, making the whole of very low resistance after firing.

Natural Silicates, as Insulators. Several natural silicates are used for various insulating purposes, a few of which are asbestos, mica and feldspar. Of these of course the most valuable by far is mica, and it is hard to imagine how the electrical industry would get along without this material. Mica for insulating purposes falls into two general groups, muscovite and phlogopite. The uses of the two micas are quite dissimilar, the former being used where highest dielectric strength and color are important and the latter where resistance to high temperature, greater resistance to abrasion as in commutators, etc., is necessary. Mica is used to a considerable extent merely split to the required thickness from the natural mineral as it occurs, but the greater part is used in what is known as "built-up" mica, this being a material made from comparatively small "splits" and a sticker of either organic or inorganic nature. Mica of the best quality is probably of the highest dielectric strength of all known materials.

Slate, soapstone and lava are used to a considerable extent for panels, bases, etc., and are very valuable owing to their toughness and ease of working to shape.

Heat Insulating Materials. In this group are the minerals asbestos, kiesulguhr, and diatomaceous earth as well as several artificial products known under the trade terms of Poplox, Calorox, Fibrox, etc., the latter products depending on mechanical and ceramic processes which leave silicates in extremely fine state of subdivision at the end of the treatment. These latter materials are becoming increasingly important, owing to the necessity of perfect heat insulation in electric stoves and other devices to secure maximum economy and efficiency. The artificial materials far surpass the natural ones.

Glass. This material, used to a considerable extent at one time for line insulation, has now practically disappeared except for very low tension lines, but very recently new glasses have been invented which, if the maker's claims are borne out, will undoubtedly bring glass into wider use again. It is understood that the properties of these new glasses are supposed to be due to a considerable percentage of boracic acid or its compounds. The introduction of the new cooking glass ware, which seems to

give satisfaction, would point towards the probability of such a glass being quite useful for certain insulations.

Glass is also used for a great variety of electrical parts such as lamp and rectifier bulbs, lamp shades, reflectors, beads, etc.

Artificial Silicates for Resistances. Many attempts have been made to make use of ceramic products for resistances for extremely heavy duty. The writer understands that a ceramic mixture, to which is added various conducting substances and the whole fired to a dense mass, is used. One may readily understand that any success with such a product would necessitate the utmost care in every operation as of course great uniformity of materials and handling would be absolutely essential in order that the manufacturer might predict the results of his mixtures. The economic production of such articles strikes the writer as the most exact factory ceramic problem of the day.

Enamels. These soft glasses are becoming more and more valuable to the industry, being used for finishes, for their diffusing properties in lighting devices, for insulated and weather-proof surfaces, as relays, resistance units and other small parts that are in exposed places and therefore should be water-proof.

Conduits. These ceramic products of the cruder sort are somewhat similar to flue linings and fire-proofing and are used to a great extent in large cities, where the use of under-ground cables, telephone wires, etc., require a permanent protective cover and spacer.

OTHER USES OF CERAMIC MATERIALS

In this division the materials are used principally as fillers or to impart characteristic properties to the product, but in no case is the formation of silicates in the process of manufacture of great importance.

Cables. Several materials commonly used in ceramic wares are used in the manufacture of rubber insulated cables. Among these are calcium oxide, and magnesia. These materials are used as fillers for retarding the action of destructive gases, etc., and play a highly important role in the efficiency of the cable. Asbestos is frequently used as a fireproof braid for wire.

Paints. Practically all ceramic materials are used in the manufacture of paints, for fillers, for their drying action on oils or other desired effects. By means of these materials the physical properties of the paints, such as the gloss, color, drying time, brushing and adhesive qualities, may be controlled. Among the common fillers are clay, barytes, oxide of zinc, dioxide of manganese and white lead.

Papers. The use of ceramic materials in this field is an indirect one but also an important one, since insulating papers are used to a great extent, and the fillers commonly used in their manufacture, such as talc and clay, enter indirectly in the insulating value of the completed material.

Molded Compounds. Many ceramic materials are used as fillers for molded compounds, each material imparting to the molding, somewhat different physical properties. Considerable quantities of these fillers are used, the commonest of which are clay, flint, feldspar, whiting, marble, glass, mica, asbestos and talc. The fillers alter the strength, finish, ease of molding and machining, color, etc., and the properties of the molded compound may in extreme cases, depend almost as much upon the filler as upon the binder. Hard rubber belongs to this group as some of the above materials are used for fillers principally to reduce the cost.

AN ATTEMPT TO FIT ENAMELS TO PLASTIC CLAY BODIES [1]

BY F. A. KIRKPATRICK, H. C. ARNOLD, AND C. F. GEIGER

The present method of enamel brick manufacture is described by Stull in Vol. XII of the *Transactions* of this society.

Enamel brick require two coats, a thick slip of high clay content, opaque when burned, and a thin glaze. The slip is applied by hand dipping or by a mechanical veneering device, the glaze by hand dipping or spraying.

The present investigation was undertaken to determine the possibility of replacing the two coats by one, an enamel, applied by a veneering device directly to a stiff plastic column issuing from a die. In this manner the cost of manufacture could be reduced considerably.

The principal defects[2] that appear after application of a slip to an enamel brick body and before burning are: 1. Pinholes, 2. Flaking, 3. Cracking.

The first named defect is generally easily overcome. Flaking and cracking, however, often cause much trouble. They are governed by two properties of the enamel: shrinkage and adhesion to the body. The control of these is the means of attack in this investigation, since the enamel is to replace the slip and must by some means be given the properties possessed by a good slip.

There are three conditions necessary for perfect mechanical fit between a slip and a plastic body, namely:

1. There must be sufficient adhesion between slip and body,

[1] A review of two theses, one by F. A. Kirkpatrick and H. C. Arnold as part of the requirement for the degree of Bachelor of Science in Ceramic Engineering and Ceramics, 1914, the other by C. F. Geiger for the degree of Bachelor of Science in Ceramic Engineering, 1915. Department of Ceramic Engineering, University of Illinois.

[2] R. T. Stull, *Trans. Amer. Cer. Soc.*, Vol. XII, p. 715.

2. The amount of shrinkage of the slip must be the same as that of the body,

3. The rate of shrinkage of the slip must be the same as that of the body.

The last two conditions may be fulfilled, but this is not enough. If adhesion is insufficient, perfect fit will not result. In many cases there is sufficient adhesion, but difference in shrinkage causes flaking, cracking, or both.

Stull[3] shows that shrinkage of the slip and adhesion of the slip to the body are controlled by change in the kind and amount of clays present, the defects of cracking and flaking being thus remedied. Also, the limits of clay content of good slips for leather hard bodies are from 25 percent to 70 percent.

The use of this method in an enamel is limited, for an enamel containing more than about 30 percent of raw clay is not glossy when burned. Also, more than 10 percent of ball clay cannot be used because its iron content imparts to the burned enamel a yellowish-brown tinge. Since raw clay in amounts up to 30 percent does not give the enamel sufficient shrinkage and adhesion, flaking and cracking result; hence it is necessary to coagulate or to increase the colloidal material in the enamel in order to obtain the proper amount of shrinkage and adhesion.

A review of ceramic literature shows that although considerable work has been done on the effect of electrolytes on clays and clay bodies in the thin-suspension and plastic states, very little is recorded on the action of electrolytes or colloidal materials on glazes.

The conditions in glaze mixtures are more complex than in clays on account of the number and amounts of different constituents in the former. This may be shown by the consideration of the conditions existing in the wet-ground enamels used in this investigation. These contain calcium carbonate, barium carbonate, zinc oxide, feldspar, tin oxide, silica as ground quartz, and clay. The carbonates and the zinc oxide are slightly soluble in water and are hydrolyzed, causing the aqueous mixture to be alkaline in nature. This would deflocculate the clay present. Reactions might occur between the adsorbed salts in the clay

[3] *Trans. Amer. Cer. Soc.*, Vol. XII, p. 715.

and the other substances in the mixture. The mineral impurities in the materials used cause further reactions. An electrolyte added to this mixture has a different effect on each substance present, making a theoretical study of the resultant effect a difficult matter.

For the purpose of regulating the shrinkage of the enamel and its adhesion to the body the following methods were used:

1. Precipitation of colloidal silicates in the enamel.
2. Coagulation by means of salts.
3. Coagulation by means of acids.
4. Coagulation by means of alkaline substances.
5. Precipitation of colloidal silicic acid in the enamel.
6. Introduction of calcium, barium, and zinc in the enamel as oxalates and addition of electrolytes.
7. Increase of clay content.
8. Addition of organic colloids.

There are certain considerations which should be noted in regard to the use of electrolytes. Iron, copper, and chromium salts, for example, would be undesirable because of the color they would impart to the burned enamel. Sulphates might cause blistering. Hydrochloric acid and the chlorides not imparting color would be desirable on account of the possibility of their forming $FeCl_3$ with the iron impurity introduced with the clay into the enamel. The $FeCl_3$ is volatilized in burning, and a whiter enamel results.

Since, in general, increase of shrinkage in ceramic mixtures is indicated by increase of viscosity of the thin aqueous suspension, the viscosity of nearly all of the mixtures used was determined. It was at first intended to use only those enamels having highest viscosity, as these would probably have highest shrinkage. After the work was under way it was decided to apply all enamels to trial pieces. The investigation thus became in part a study of the effect of electrolytes on viscosity of the enamels, and of the relation of viscosity to shrinkage and adhesion.

The enamels were ground wet in ball mills for two hours and passed through a 100 mesh screen. The percent of water

was determined in a small sample. Enough enamel was weighed into each of a series of graduated cylinders to give 50 grams of solid material. The electrolytes were introduced and enough water was added to bring the total volume to 100 cc. The cylinders were then shaken by hand for three minutes each.

The viscosity of the enamels was next determined by means of an Ostwald viscosimeter having an efflux opening of two millimeters diameter. The viscosimeter was standardized by determining the time of flow of distilled water. Each viscosity value is the average of several, usually three, determinations. The first tests were made immediately after the addition of electrolytes, unless otherwise stated.

The numerical values of relative viscosity range from 1 to 200. The probable experimental errors in these values as determined from variations in results are roughly as follows:

Relative Viscosity.	*Probable Error*
1 to 5	1 percent to 2 percent
5 to 10	2 percent to 10 percent
10 to 200	10 percent to 20 percent

The largest errors in the high viscosity values are probably due to segregation in the viscosimeter, the solid material settling to the bottom causing increase in time of flow.

As shown by Schmidt and Jones,[4] an error of 1 or 2 percent is introduced on account of the comparison of two substances (in our case water and slip) having large differences in time of flow. The error is eliminated by using several viscosimeters with efflux tubes of increasing diameters and calibrating the viscosimeters against each other.

Schmidt and Jones give the following formula as that ordinarily used in calculating relative viscosity from determinations using the Ostwald viscosimeter:

[4] *Amer. Chem. Jour.*, Vol. 42, 1909, pp. 64 and 65.

$$\frac{n_1}{n_2} = \frac{r_1 t_1}{r_2 t_2}$$

Where n = viscosity coefficient,
r = density,
t = time of flow.

The formula used in this investigation is:

$$\frac{n_1}{n_2} = \frac{t_1}{t_2}$$

In order to obtain the correct viscosity, our results should all be multiplied by the density of the enamel slips. The density of the mixtures was not determined, but for a fresh slip it was calculated to be about 1.25, but this value, in all probability, would increase with increasing coagulation.

Since a study of viscosity was not the main object of the work, no special effort was made to eliminate the errors in mixtures of high viscosities.

After the viscosities had been determined, the enamels were brought to a suitable dipping consistency by evaporation or dilution and then applied, by dipping or pouring, to stiff-mud trial pieces one and one-half inches square. These were air dried, and those that had no defects were burned to cone 7 in a down-draft oil-kiln.

The test pieces had the following compositions:

1. 100 percent St. Louis fire clay,
2. 70 percent St. Louis fire clay and 30 percent 20 mesh fire brick grog,
3. 100 percent Olive Hill plastic fire clay,
4. 70 percent Olive Hill plastic fire clay and 30 percent grog,
5. 70 percent Pennsylvania fire clay and 30 percent grog,
6. 70 percent Bloomingdale stoneware clay and 30 percent grog,

7. 70 percent Templeton, Pa., fire clay and 30 percent grog.

Every defect of fit was carefully noted. All the results cannot be given, but a few are shown on the viscosity diagrams by the following abbreviations placed along the curves:

Fo = Enamel flaked, extending over edge of trial pieces, or buckled, indicating too small shrinkage of the enamel.
F = Enamel flaked with same shrinkage as trial piece.
C = Enamel cracked and flaked. Impossible to determine relative shrinkage.
G = Enamel had sufficient adhesion and proper shrinkage. Slight defects in some cases.

The following are the formulæ of some of the enamels used; their maturing range is from cone 7 to cone 9:

Enamel 25. Containing 25 percent of raw clay.

$$\left.\begin{matrix} .10\ K_2O \\ .35\ CaO \\ .20\ BaO \\ .35\ ZnO \end{matrix}\right\} .64\ Al_2O_3 \left\{\begin{matrix} 3.80\ SiO_2 \\ .12\ SnO_2 \end{matrix}\right.$$

Batch Weights

Brandywine Summit feldspar	55.8
Whiting	35.0
Barium carbonate	39.4
Zinc oxide	28.4
Ohio 8 hour flint	127.2
Tin oxide	18.0
Pike's No. 20 Eng. ball clay	44.0
M. G. R. Eng. china clay	66.0
M. G. R. Eng. china clay, calcined	25.3
	439.1

Enamel 35. Containing 35 percent of raw clay.

$$\left.\begin{matrix} .40\ CaO \\ .25\ BaO \\ .35\ ZnO \end{matrix}\right\} .64\ Al_2O_3 \left\{\begin{matrix} 3.80\ SiO_2 \\ .12\ SnO_2 \end{matrix}\right.$$

Batch Weights

Whiting	40.0
Barium carbonate	49.3
Zinc oxide	28.4
Ohio 8 hour flint	151.2
Tin oxide	18.0
Pike's No. 20 Eng. ball clay	45.0
M. G. R. Eng. china clay	119.0
	450.9

Enamel Ox-25. Containing 25 percent of raw clay.

$$\left.\begin{matrix} .10\ K_2O \\ .35\ CaO \\ .20\ BaO \\ .35\ ZnO \end{matrix}\right\} .64\ Al_2O_3 \left\{\begin{matrix} 3.80\ SiO_2 \\ .12\ SnO_2 \end{matrix}\right.$$

Batch Weights

Brandywine Summit feldspar	55.8
Calcium oxalate	44.8
Barium oxalate	45.0
Zinc oxalate	53.7
Ohio 8 hour flint	127.2
Tin oxide	18.0
Pike's No. 20 Eng. ball clay	48.0
M. G. R. Eng. china clay	72.0
M. G. R. Eng. china clay, calcined	16.2
	480.7

RESULTS

Precipitation of Colloidal Silicates in the Enamel. Sodium silicate solution was first added to the wet enamels and then magnesium chloride solution in the proportion of 65 percent sodium silicate to 35 percent magnesium chloride by weight. This ratio was chosen, because out of nine mixtures, it gave the largest amount of precipitate of magnesium silicate.

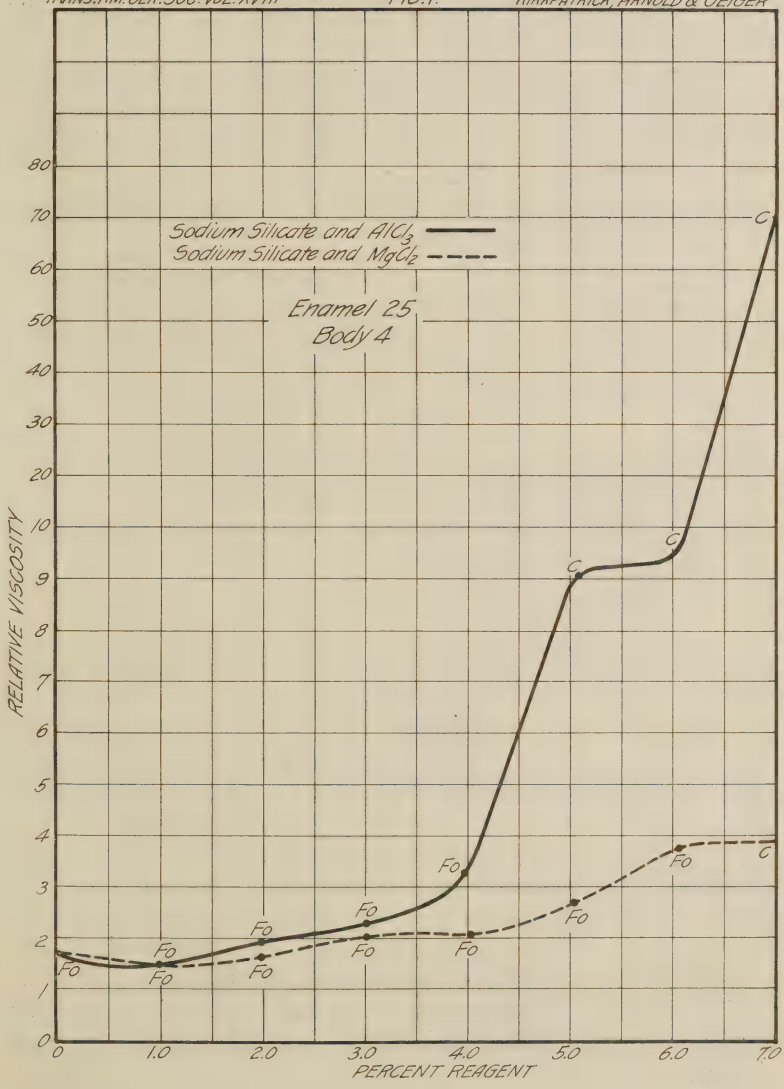
The same process was carried out using sodium silicate and aluminium chloride, the same percentages as above being employed.

The viscosity curves are shown in Fig. 1. Decrease of viscosity occurred at first, probably because of excess of sodium silicate. With larger amounts of reagent, viscosity increased. When applied to trial pieces, the enamels had too little shrinkage and flaked off in drying.

Coagulation by Means of Salts. The salts used were: $CaCl_2$, $ZnCl_2$, $MgSO_4$, potassium alum, [K_2SO_4, $Al_2(SO_4)_3 \cdot 24 H_2O$], $Al_2(SO_4)_3$, $AlCl_3$, $SnCl_4$. These furnish univalent, bivalent, trivalent, and quadrivalent cations. By use of these compounds the effect of valence on the viscosity of the enamels was observed.

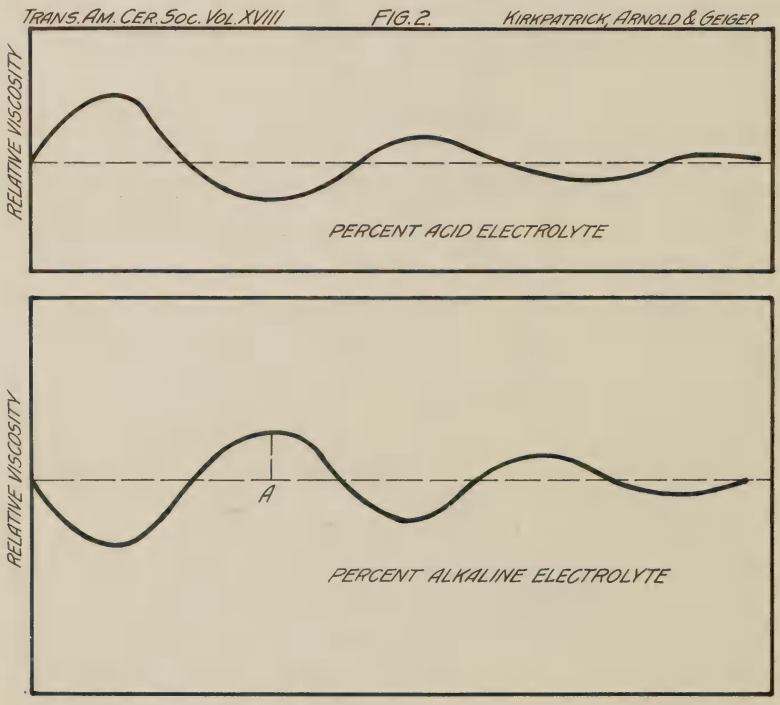
The theoretical viscosity curves for addition of electrolytes to clay slips are shown in Fig. 2. They are based on considerations found in Freundlich's "Kapillarchemie". The character of these curves would lead us to expect maximum and minimum points in viscosity curves for enamels, although deviations would probably occur because of the various chemical reactions in the complex mixture.

Viscosity curves for additions of salts, Fig. 3, do show the existence of these maximum and minimum points. All of the samples stood 24 hours before being tested. It is noted that the bivalent ions from $MgSO_4$ and $CaCl_2$ gave the least increase in viscosity, the trivalent ion from $Al_2(SO_4)_3$, $AlCl_3$, or alum gave greater viscosity, while the quadrivalent ion from $SnCl_4$ gave the greatest viscosity. $ZnCl_2$ is an exception to the rule, ranking with trivalent and quadrivalent ions. In this case, however, the high viscosity may be due to a Sorel cement reaction between


TRANS. AM. CER. SOC. VOL. XVIII
FIG. 1.
KIRKPATRICK, ARNOLD & GEIGER
Sodium Silicate and $AlCl_3$
Sodium Silicate and $MgCl_2$
Enamel 25
Body 4
RELATIVE VISCOSITY
PERCENT REAGENT
0
1.0
2.0
3.0
4.0
5.0
6.0
7.0
10
20
30
40
50
60
70
80
Fo
C

$ZnCl_2$ and the ZnO of the enamel. Although this reaction might increase viscosity, it did not cause the enamel to adhere to the body, as all enamels flaked or cracked on the trial pieces. The remaining salts gave no better results.

Coagulation by Means of Acids. The acids employed were hydrochloric, oxalic, and boric. After being shaken, the wet enamels gave alkaline reaction toward litmus, indicating that



the acids had been neutralized by the bases in the enamel. However, hydrochloric acid caused the enamel to be twenty times as viscous as the original sample, oxalic acid caused it to be four times as viscous, and boric acid caused it to be only slightly more viscous.

The increase may be due in part to the products of the reaction between the acids and bases. From the use of hydro-

chloric acid would result chlorides and carbonic acid. It was determined that calcium chloride alone in amounts up to 3 percent had very little effect on the viscosity of the enamel and that zinc chloride greatly increased viscosity. Ashley gives barium chloride as a coagulator of clay suspensions.[5] A part of the increase is probably due to hydrogen ion being taken up and retained by the clay, causing coagulation.

When the enamels to which acid had been added were dried on test pieces they flaked and cracked badly so that it could not be determined whether shrinkage was correct or not. The results are shown in Fig. 4.

Coagulation by Means of Alkaline Substances. The theoretical viscosity curve for additions of alkaline substances to clay has been given (Fig. 2.) Similarly if amount A of alkali were added to an enamel, viscosity might be greater than that of the original.

The alkaline substances used were sodium carbonate and borax. The viscosity curves are shown in Fig. 5. It is noted that the amount of reagent required to produce coagulation would be too great for practical use of the method. None of the enamels had sufficient shrinkage or adhesion, and all flaked off the trial pieces.

Precipitation of Colloidal Silicic Acid in the Enamel. Colloidal silicic acid was precipitated in the enamel by first adding sodium silicate solution and then introducing hydrochloric acid or oxalic acid. A solution of sodium silicate (90 grams of $Na_2O{\cdot}2.5\ SiO_2$ per liter) was mixed with a solution of hydrochloric acid (18.2 grams per liter) in various proportions. That mixture containing 80 percent of sodium silicate set in three minutes to a jelly, a shorter time than that required for any other percentages. Hence this proportion was used for addition to the enamel.

The same procedure was followed using sodium silicate and oxalic acid, the strength of each solution being 90 grams per liter. The mixture containing 80 percent by weight of sodium silicate set at once, and in one hour the 70 percent mixture set. The former proportion was employed.

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[5] *U. S. Bur. Stand. Bull. 23*, p. 74.

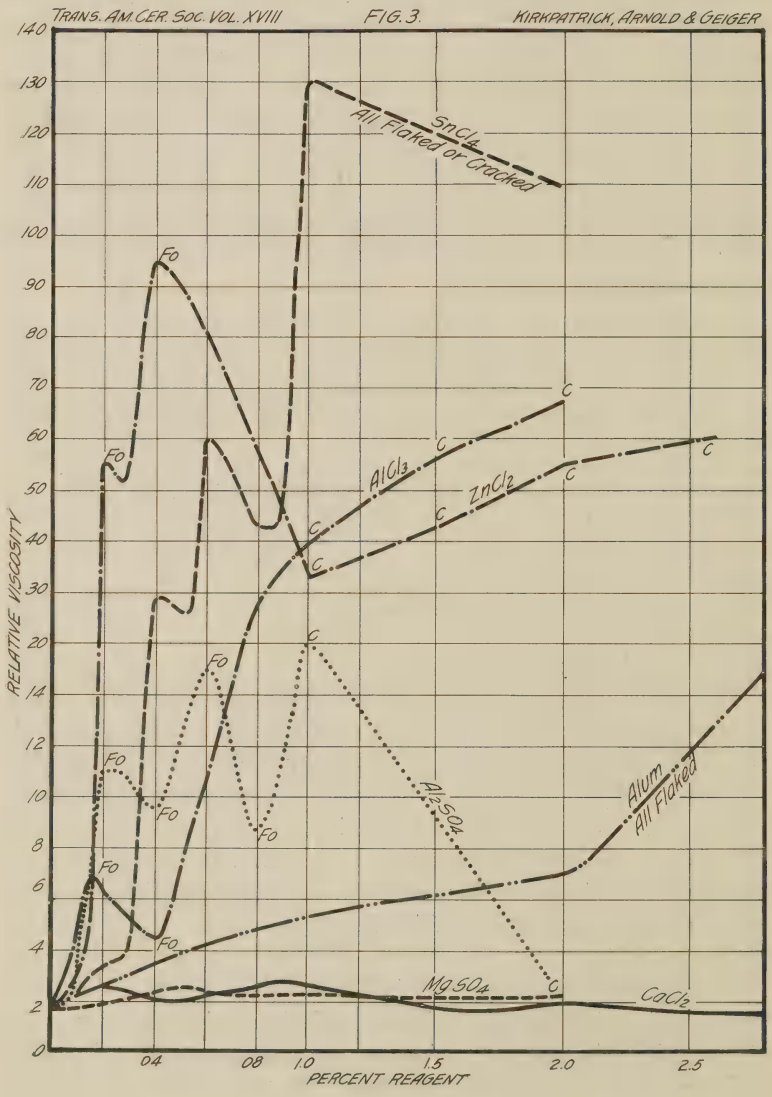
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FIG. 3.
KIRKPATRICK, ARNOLD & GEIGER
RELATIVE VISCOSITY
PERCENT REAGENT
SnCl4
All Flaked or Cracked
AlCl3
ZnCl2
Al2SO4
Alum
All Flaked
MgSO4
CoCl2
Fo
C
0
2
4
6
8
10
12
14
20
30
40
50
60
70
80
90
100
110
120
130
140
04
08
1.0
1.5
2.0
2.5

Two of the viscosity curves for additions of silicic acid are shown in Fig. 6. With additions of reagent up to 1 or 2 percent there was a decrease in viscosity due probably to the excess sodium silicate and to the sodium salts formed by the reaction, the mixture added being alkaline to litmus. With larger amounts of reagents, the silicic acid increased the viscosity, overcoming the effect of the deflocculating salts. At about 4 or 5 percent the enamels thickened greatly, some setting to a jelly. When hydrochloric acid was employed as the precipitating agent, the enamels flaked with all amounts, indicating too little shrinkage and adhesion. When oxalic acid was used, shrinkage and adhesion were good with 4, 5, and 6 percent of reagent.

When the foregoing method is used there are likely to be present in the enamel sodium oxalate and an excess of sodium silicate, both of which decrease viscosity. Silicic acid, which had been washed by decantation to remove these salts, was added to another series of enamels, but flaking and cracking resulted with all amounts used.

In connection with this method Mr. Stull has suggested the following possible procedure in the manufacture of enamel brick: Just enough sodium silicate is added to the enamel to give minimum viscosity. The minimum amount of water is then necessary for grinding and screening. The enamel is placed in a blunger and more sodium silicate and enough oxalic acid are added to thicken the enamel sufficiently for application. This eliminates filter-pressing and repugging.

This scheme was attempted in the laboratory, but the enamel cracked because of too high shrinkage. The amounts of reagent used were large, 4 percent sodium silicate and 1 percent oxalic acid, because these amounts seemed to be necessary to obtain adhesion.

There seems to be no reason, however, why the method may not be employed with an enamel or slip that already has perfect fit, since the amounts of reagent could be kept below 1 percent.

Introduction of Calcium, Barium, and Zinc, in the Enamel as Oxalates and Addition of Electrolytes. Addition of these elements as oxalates instead of carbonates, it was hoped, would eliminate chemical reactions due to the alkaline nature of water

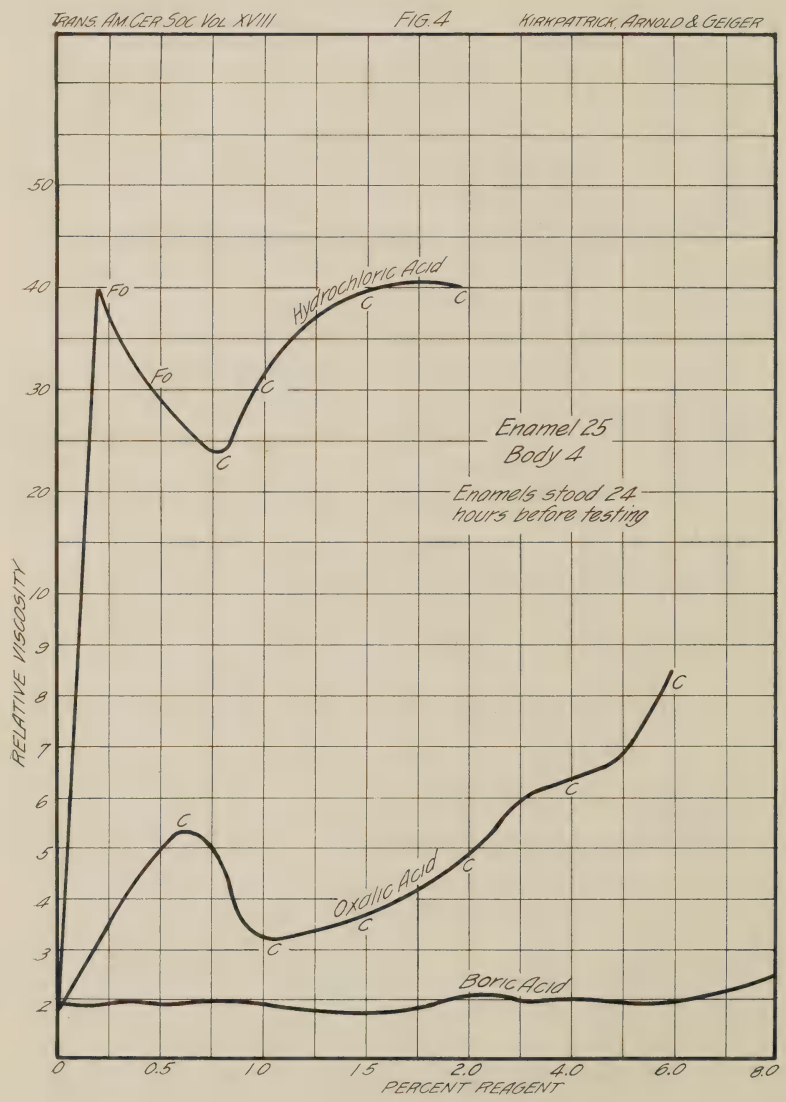
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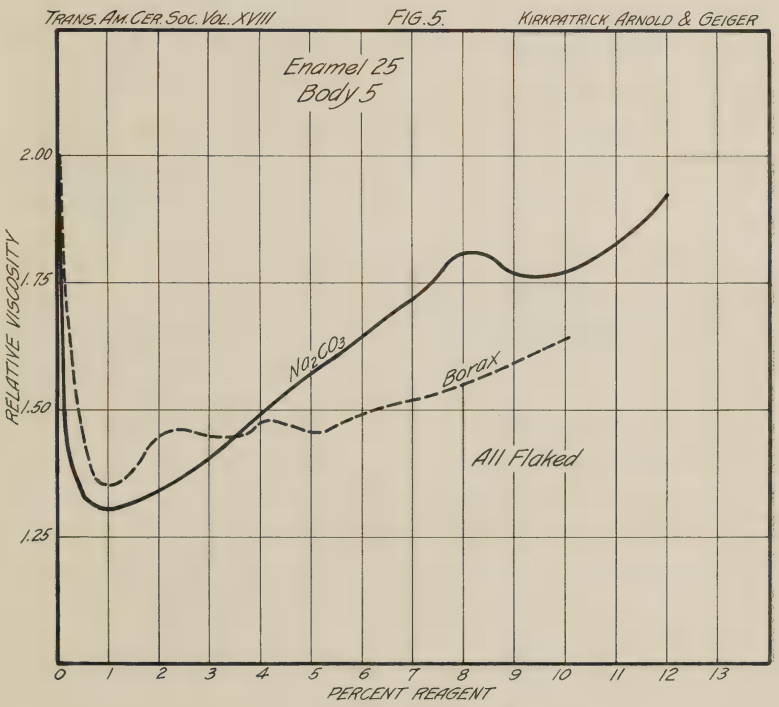
FIG. 4
KIRKPATRICK, ARNOLD & GEIGER
RELATIVE VISCOSITY
PERCENT REAGENT
Hydrochloric Acid
Oxalic Acid
Boric Acid
Enamel 25
Body 4
Enamels stood 24 hours before testing
Fo
C

in contact with carbonates. The enamel prepared in this manner gave an acid reaction with litmus owing to hydrolysis of the slightly soluble oxalates with formation of oxalic acid. This enamel, with no electrolytes added, had a relative viscosity twice that of the enamel containing the carbonates.

The oxalate enamel was treated with the following reagents in the same manner as the carbonate enamel: oxalic acid, so-



dium silicate, hydrochloric acid, sodium silicate and hydrochloric acid, sodium silicate and oxalic acid.

The viscosity curves are shown in Fig. 7. They require no detailed discussion. The oxalate enamels adhered to the trial pieces, on drying, much better than did the carbonate enamels and gave a much larger percentage of perfect trial pieces. Fig. 8 shows the viscosity curve for oxalic acid in enamel Ox. 15.

This curve approaches closely the theoretical curve shown in Fig. 2.

Increase of Clay Content. All of the results reported under other methods were obtained with an enamel containing 25 percent of raw clay. However, many of the series of determinations were duplicated with enamels containing 15 percent and 35 percent.

The results in the latter cases were little different from those obtained with the former except in one instance, that of additions of sodium silicate and oxalic acid in the 35 percent raw clay enamel. This enamel containing 3, 4, and 5 percent of reagent gave perfect fit on St. Louis and Olive Hill clay and grog bodies, while the 15 percent and 25 percent raw clay enamels with the same reagents gave a smaller number of good trial pieces.

Addition of Organic Colloids. The organic colloids used were starch, gum tragacanth, and glue.

A known weight of starch was boiled with water to a paste and added to a large batch of enamel; the mixture was ground in a ball mill. A series of enamels was prepared by blending this and the original enamel. No viscosity determinations were made as it was believed that this factor would increase approximately in direct proportion to the amounts of starch contained. The enamels were applied in a pasty condition.

Additions of one percent starch produced adherence with the Templeton and Bloomingdale bodies, but three percent was required for the St. Louis body. The trial pieces were burned to cone 7½ and all the coatings held well. The enamel, which was thick enough to hide the body completely, was not very smooth, but this could be remedied by change in composition or temperature of burning.

A gum tragacanth series was prepared in the same manner. Amounts of 0.3 to 0.5 percent produced adequate adhesion in drying, and the trials withstood the fire without flaking or crawling even though they were placed on edge during burning. The indications were that amounts of 0.8 percent or more were to be avoided.

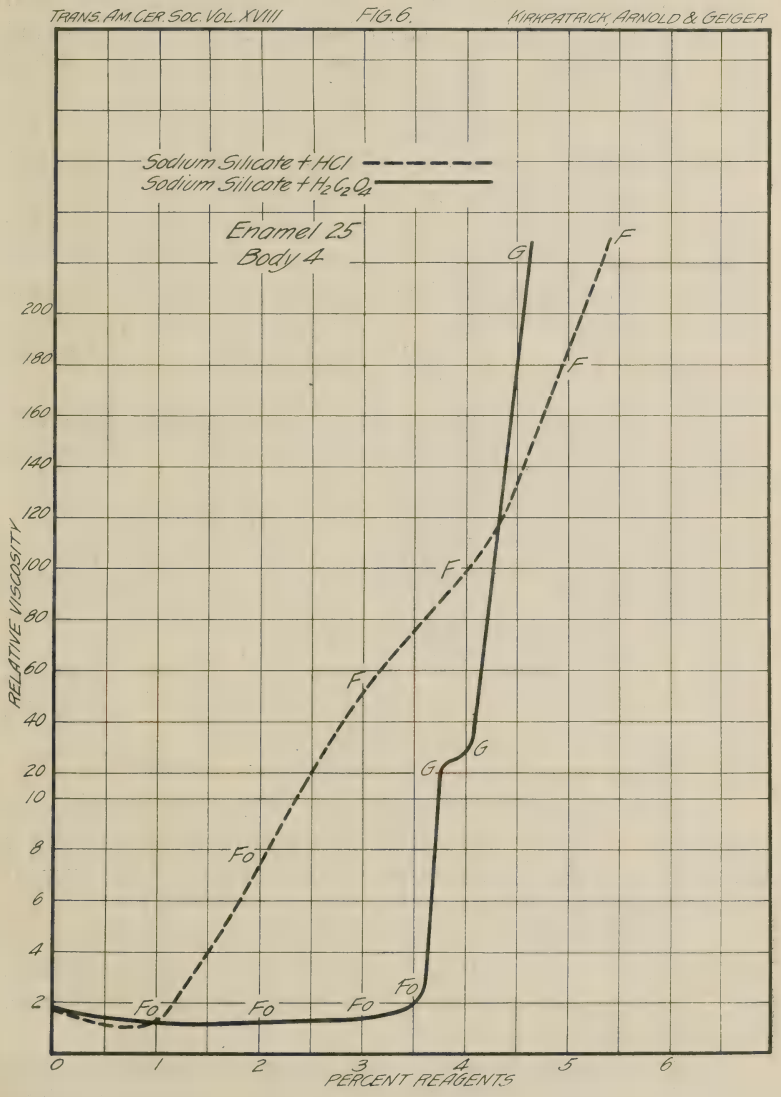

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FIG. 6.
KIRKPATRICK, ARNOLD & GEIGER
Sodium Silicate + HCl
Sodium Silicate + $H_2C_2O_4$
Enamel 25
Body 4
RELATIVE VISCOSITY
200
180
160
140
120
100
80
60
40
20
10
8
6
4
2
0
1
2
3
4
5
6
PERCENT REAGENTS
F
G
Fo

Liquid glue was diluted with water, and enough of this solution was added to enamels to bring in amounts of from 1 to 6 percent of the original. The mixtures were very sticky and were applied thickly. On all pieces the enamel coating flaked off without cracking. The flaked piece was hard and longer than the dried body showing too little shrinkage in the enamel.

Time Effect. A number of investigators have observed that the viscosity of some wet ceramic mixtures, to which electrolytes have been added, changes with time thus destroying their usefulness. Hence, in the present investigation considerable attention was paid to the time effect of reagents and in some of the series, viscosity tests of the same enamels were made every day for one or two weeks. Some of the effects observed using enamel 25 were as follows:

(1) Additions of $SnCl_4$: viscosity increased with time with all amounts, the larger the amount used the greater the increase. Total time of test, 120 hours.

(2) Additions of $MgSO_4$: all amounts of electrolyte gave increase in viscosity up to 24 hours after which they gave decrease in viscosity. The changes were not great, varying from 1.1 to 1.6. Forty grams of solid material were used in the tests instead of 50 grams. Total time, 78 hours.

(3) Additions of alum: amounts of 1 percent and higher effected very great increase in viscosity with time, all samples being too thick to flow after 144 hours. Samples containing less than 1 percent changed very little with time.

(4) Additions of sodium silicate and oxalic acid: amounts up to 3 or 4 percent showed little change with time, 5 and 6 percent caused the enamel to pass from a state of thin suspension to a jelly in 24 hours.

(5) Additions of sodium silicate and hydrochloric acid: amounts up to 1 and 2 percent produced little change with time; 3 to 6 percent gave continuous decrease in viscosity with time. Total time, 48 hours.

(6) Additions of $AlCl_3$: amounts up to 0.06 percent produced little change in viscosity with time. Percentages from 0.08 to 2 gave great increase in 24 hours, but after two weeks, viscosity decreased approaching the original values.

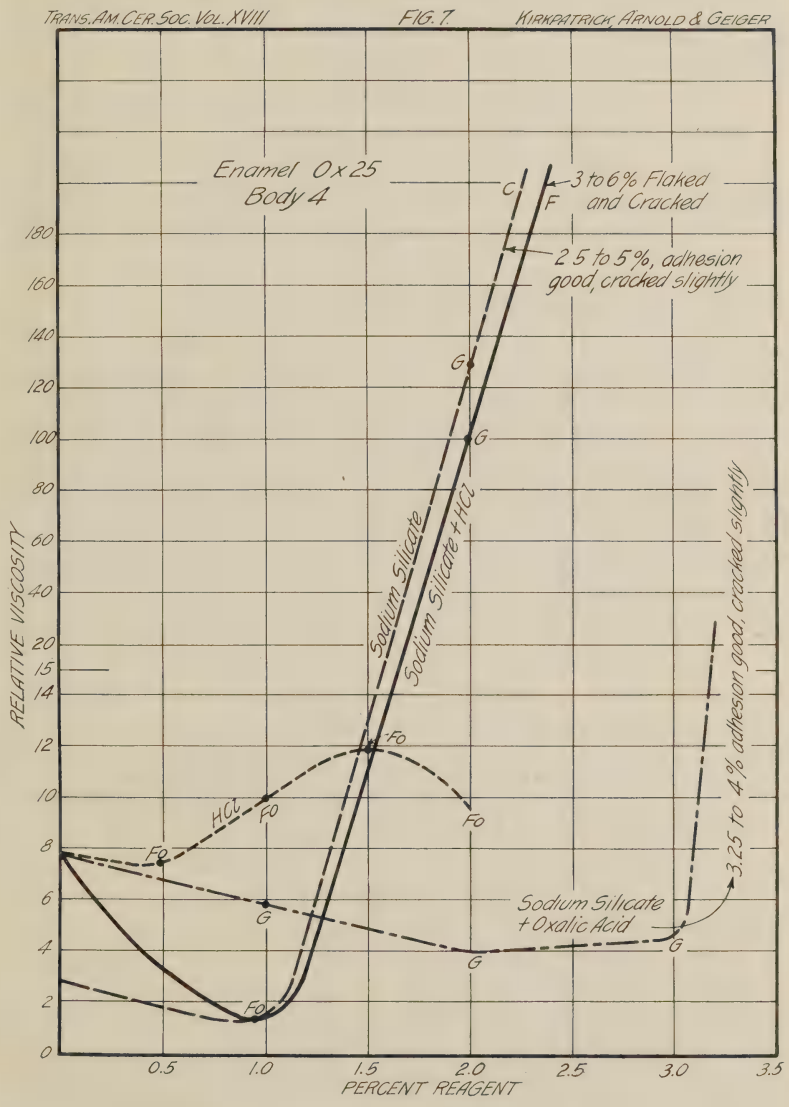
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FIG. 7.
KIRKPATRICK, ARNOLD & GEIGER
Enamel 0x25
Body 4
3 to 6% Flaked and Cracked
2.5 to 5%, adhesion good, cracked slightly
Sodium Silicate
Sodium Silicate + HCl
Sodium Silicate + Oxalic Acid
3.25 to 4% adhesion good, cracked slightly
HCl
C
F
G
Fo
RELATIVE VISCOSITY
PERCENT REAGENT
0
2
4
6
8
10
12
14
15
20
40
60
80
100
120
140
160
180
0.5
1.0
1.5
2.0
2.5
3.0
3.5

(7) Additions of HCl: Amounts up to 0.1 percent gave little change in viscosity with increasing time: 0.1 percent to 1.5 percent gave decrease in viscosity with time. Duration of test, 48 hours.

(8) Additions of oxalic acid: All amounts gave considerable increase of viscosity in 24 hours.

(9) Additions of washed silicic acid: Amounts from 0.1 to 0.7 percent gave no change in viscosity with time, 0.8 to 1.0 percent gave considerable increase after ten days.

From these results it is seen that no general rule can be stated in regard to time effect in the enamel used.

If it were known which constituents of the enamel cause the time effect, they might be replaced by other forms of the substances. This was determined by making viscosity tests of aqueous suspensions of each enamel constituent with no electrolyte and with 3 percent potash alum. The determinations were made at intervals for a period of two weeks. Mixtures of 25 grams of solid per 100 cc. of suspension gave too small changes to afford good comparative results. Mixtures containing 30 g. solid per 100 cc. gave comparative results which are shown in Fig. 9. Mixtures containing 50 g. solid per 100 cc. gave only partially comparative results since some of them were too thick to flow at the start and were diluted. Changes in the 30 g. mixtures are comparable with those containing 40 to 50 grams, the curves in the two series being approximately parallel.

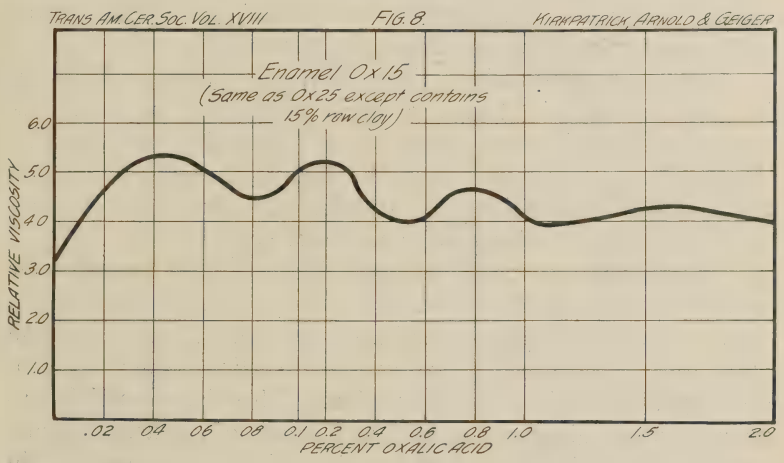
From Fig. 9 it is seen that the viscosity of the following mixtures changed very little with time:

English ball clay,
English china clay,
English china clay with alum,
Whiting ($CaCO_3$),
Whiting with alum,
Tin oxide (SnO_2) with alum,
Flint (SiO_2 as ground quartz),
Flint with alum,
Potash feldspar,

Potash feldspar with alum,
Enamel 25,
Barium carbonate ($BaCO_3$) with alum.

The viscosity of the following mixtures increased greatly with time, some thickening sooner than others:

Zinc oxide (ZnO) with alum,
Zinc oxide,
Tin oxide (SnO_2),
English ball clay with alum,
Enamel 25 with alum,
Barium carbonate ($BaCO_3$).



It is evident that change in chemical form of constituents to prevent change of viscosity with time would be difficult if not impossible. The enamel should be used before the change takes place.

It is beyond the scope of this paper to attempt to explain in detail the results shown in Fig. 9. A few points may be noticed in passing. The changes in viscosity are due to the slow speed of some chemical reactions. We note that ZnO, SnO_2, or $BaCO_3$ thicken although no electrolyte is added, but when mixed with the other constituents in the enamel, their effect is

neutralized, and no change occurs in the viscosity of the enamel. SnO_2 thickens alone, but addition of alum entirely neutralizes the high viscosity. This is true also of $BaCO_3$. It is possible that SnO_2 dissolves slightly forming stannous hydroxide, $Sn(OH)_2$. If the SnO_2 particles possessed positive electric charges, the negative charges from OH ions from $Sn(OH)_2$ would neutralize the charges on the particles causing coagulation. Addition of alum would give an excess of H ions which would neutralize the OH ions and repel the SnO_2 particles causing deflocculation and decrease viscosity.

$BaCO_3$ is slightly soluble in water with alkaline reaction. If the $BaCO_3$ particles possessed positive electric charges, these would be neutralized by the negative charges on the OH ions causing coagulation and increase in viscosity. Addition of the acidic alum solution would neutralize this effect.

ZnO probably dissolves slightly forming H_2ZnO_2, which ionizes more as an acid than as a base. If the ZnO particles possessed negative electrical charges, these would be neutralized by the charges on the H ions from the H_2ZnO_2 causing coagulation and increase in viscosity. On addition of alum the H ion concentration would be increased and viscosity increased.

Explanations of single reactions, however, will not solve the problem of the equilibria involved in the enamel slip. When the seven or eight enamel constituents are mixed with water and alum added, there may be a hundred reactions gradually proceeding to a condition of equilibrium. The above possible explanations are given more for the purpose of arousing discussion on the subject than with the idea that they would aid in solving the problem of the cause of time effect.

SUMMARY.

Method 1. Precipitation of colloidal silicates in the enamel. This method was not successful. The magnesium and aluminium silicates gave increase in viscosity, but gave no bond, owing to the fact that these silicates dry to impalpable powders, weakening the natural clay bond by dilution.

Method 2. Coagulation by means of salts. This method gave no satisfactory results. The salts used, $Al_2(SO_4)_3$, $AlCl_3$,

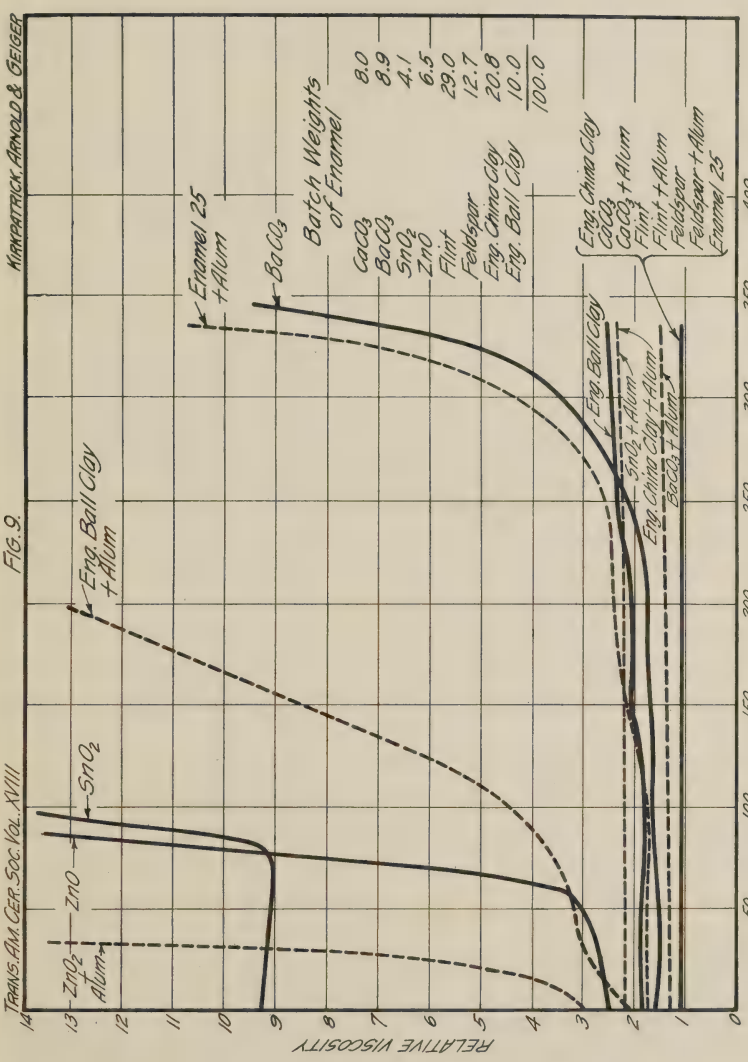

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FIG. 9.
KIRKPATRICK, ARNOLD & GEIGER
RELATIVE VISCOSITY
TIME IN HOURS
Batch Weights of Enamel
CaCO3 8.0
BaCO3 8.9
SnO2 4.1
ZnO 6.5
Flint 29.0
Feldspar 12.7
Eng. China Clay 20.8
Eng. Ball Clay 10.0
100.0
Enamel 25 +Alum
BaCO3
Eng. Ball Clay +Alum
SnO2
ZnO
ZnO2 +Alum
Eng. Ball Clay
SnO2 +Alum
Eng. China Clay +Alum
BaCO3 +Alum
Eng. China Clay
CaCO3
CaCO3 +Alum
Flint
Flint +Alum
Feldspar
Feldspar +Alum
Enamel 25

$ZnCl_2$, $CaCl_2$, $SnCl_4$, $MgSO_4$, and alum, increased viscosity to a greater or less degree but did not afford adhesive power.

Method 3. Coagulation by means of acids. The results were practically the same as those obtained with salts. The acids used were HCl, H_3BO_3, and $H_2C_2O_4$.

Method 4. Coagulation by means of alkaline substances. Borax and Na_2CO_3, known to increase adhesion of glazes and slips to bodies with little or no shrinkage, did not improve the fitting qualities of the enamels used. Shrinkage of the enamel was too small and flaking resulted.

Method 5. Precipitation of colloidal silicic acid in the enamel. This method gave more promising results than previous methods. Enamels containing 25 percent and 35 percent raw clay, and treated with 3 to 5 percent of sodium silicate and 1 to 2 percent of oxalic acid fitted the stiff-mud bodies well. The adjustment of shrinkage of enamels thus treated must be made accurately, perhaps too accurately for application in many cases. What is required is a range rather than a point of adaptability of enamel to body.

It is possible that the proportion of electrolytes used, (80 percent sodium silicate and 20 percent acid) is not best suited for the purpose, even though the mixture solidified more quickly than any other. Silicic acid from any of the mixtures would set when the enamel dried. The properties of shrinkage and adhesion would be dependent upon the amount of excess sodium silicate or of acid.

Washed silicic acid was of no value in producing adhesion, hence it is possible that in the use of this method, adhesion is produced by means of the salts present, while the shrinkage is due to the silicic acid.

Method 6. Introduction of calcium, barium, and zinc in the enamel as oxalates, and addition of electrolytes. This method in general gave a larger percentage of perfect trial pieces with all chemicals tried than did methods using enamels containing calcium and barium as carbonates and zinc as the oxide. The adhesion afforded by the oxalates may be due to crystallization. The present price of these oxalates, however, would probably prevent their use in such amounts as required in the enamel.

Method 7. Increase of raw clay content. Increasing the raw clay content from 15 percent to 35 percent gave better fitting enamels in only one case, that of the addition of sodium silicate and oxalic acid. This however, might be used to advantage in some instances.

Method 8. Addition of organic collooids. This method was the only one found to be readily applicable in practice. The addition of starch paste or gum tragacanth to the enamel gives the much desired range of fit to bodies of different composition and shrinkage; even this method, however, is not of universal application.

Treatment of body. Another method should be mentioned, that of decreasing shrinkage of the clay body by addition of grog or of electrolytes. Although not attempted in this investigation, there seems to be no reason why the method should not aid in fitting enamels to stiff-mud bodies.

Time effect. Change in viscosity with time is due to slow rate of chemical reactions in the enamel slip. The specific causes of time effect were not determined and no general rules regarding time effect in the enamel used can be stated.

The time effect was determined for the enamel using different electrolytes, and for each enamel constituent with 3 percent potash alum and also with no electrolyte.

GENERAL CONCLUSIONS

Ashley[6] has discussed the regulation of glazes and engobes by control of the colloidal materials. The results of the present investigation show that the regulation referred to is not so easy of accomplishment as his statements indicate; in fact it is very difficult.

Ashley[7] states that coagulated gels act as adhesives. This statement should be modified. Coagulated gels very probably do act as adhesives, but if there is a difference of shrinkage between the glaze and the body, the glaze will flake off in drying despite its adhesive power.

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[6] *Bull. 23, U. S. Bur. Stand.*, pp. 102-108.
[7] *Ibid.*, p. 103.

In general, shrinkage of the enamel increases with increase of viscosity, but there are many exceptions due to chemical reactions in the complex mixture. Viscosity of the enamel gives no indication of adhesive power.

The results indicate that adhesion is governed in some cases by other than a colloidal or sticky condition of the enamel: perhaps by crystallization of the substances added or of products of reactions.

The two properties of the enamel, shrinkage and adhesion, are not well understood and will not be until we study them more thoroughly from a physical-chemical standpoint.

Of all substances used, starch and gum tragacanth were most effective in producing perfect fit between enamel and body both in drying and burning. The sodium silicate-oxalic acid additions also gave very promising results.

In conclusion, the writers wish to acknowledge their indebtedness to Mr. R. T. Stull, who gave many valuable suggestions during the entire course of the investigation.

METHODS OF CALCINING CLAY FOR GROG

BY C. B. HARROP.

Non plastic clay is an ingredient of a great many of our cruder and heavier clay wares, such as furnace refractories, glass house and gas house refractories, tanks and retorts for metallurgical distillation processes, terra cotta, saggers, etc. This nonplastic clay may be ground and used in the mix in the raw state; it may be calcined and then crushed, or, if a clay of some plasticity is used, it may be ground, shaped into blocks by the mud process, burned and then crushed.

The principal reason for calcining a clay is to effect the greater part of its shrinkage before it is made up into the permanent shape. Incidentally the mechanical and chemical water is eliminated and the carbonaceous matter is oxidized. The purposes of a non-plastic clay or a grog in a refractory body are two-fold, — first, to increase the refractoriness of the ware by forming a skeleton, which may remain erect even after the bond clay begins to soften; second, to produce a less dense body and thereby reduce the chance of damage on account of temperature changes.

In general the finer the ingredients of a body and the more homogeneous the mixture, the less refractory will it be, while on the other hand, the coarser the nonplastic clay in a body (keeping within practical limits) the more refractory will it be.

It is common practice at some fire brick factories to crush the bats and culls for use as grog, instead of using flint clay or plastic clay that has been calcined to a high temperature. Even supposing that the bats contain the standard proportions of plastic and flint or nonplastic clay, if they are crushed and used in a new mix, being added as so much percent by weight of flint or nonplastic clay, it is evident that there will be an excess of bond clay with the result that the ware will not be so refractory.

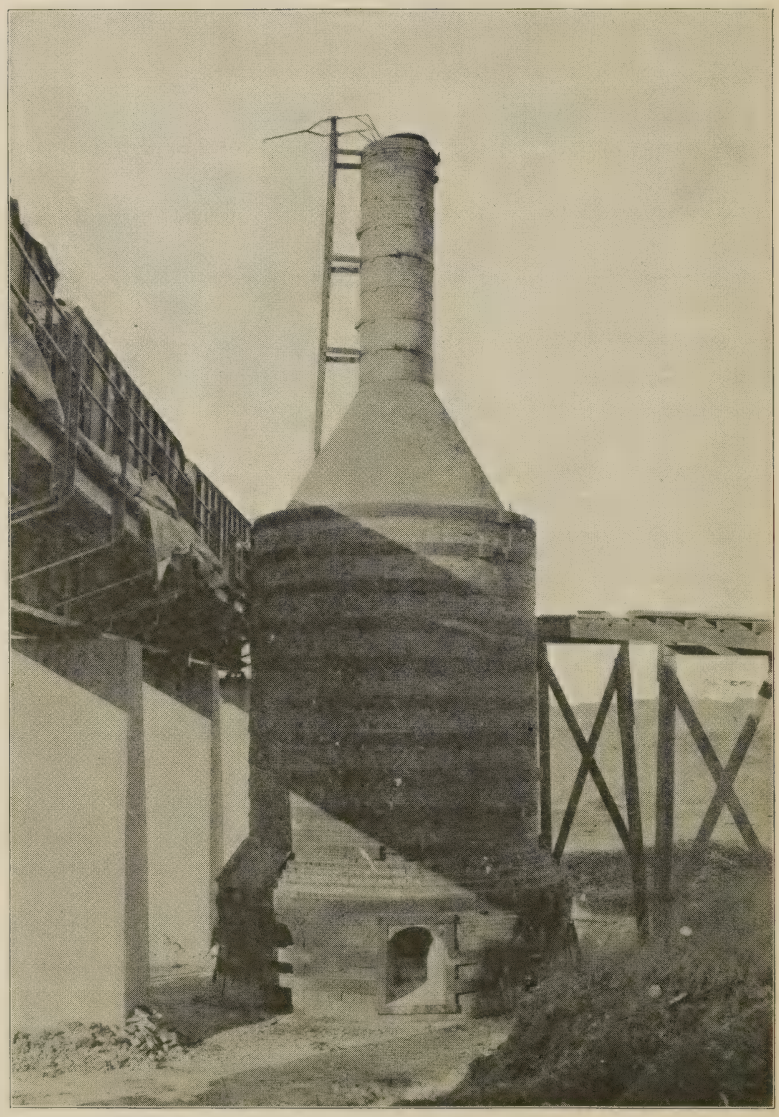


FIG. 1.

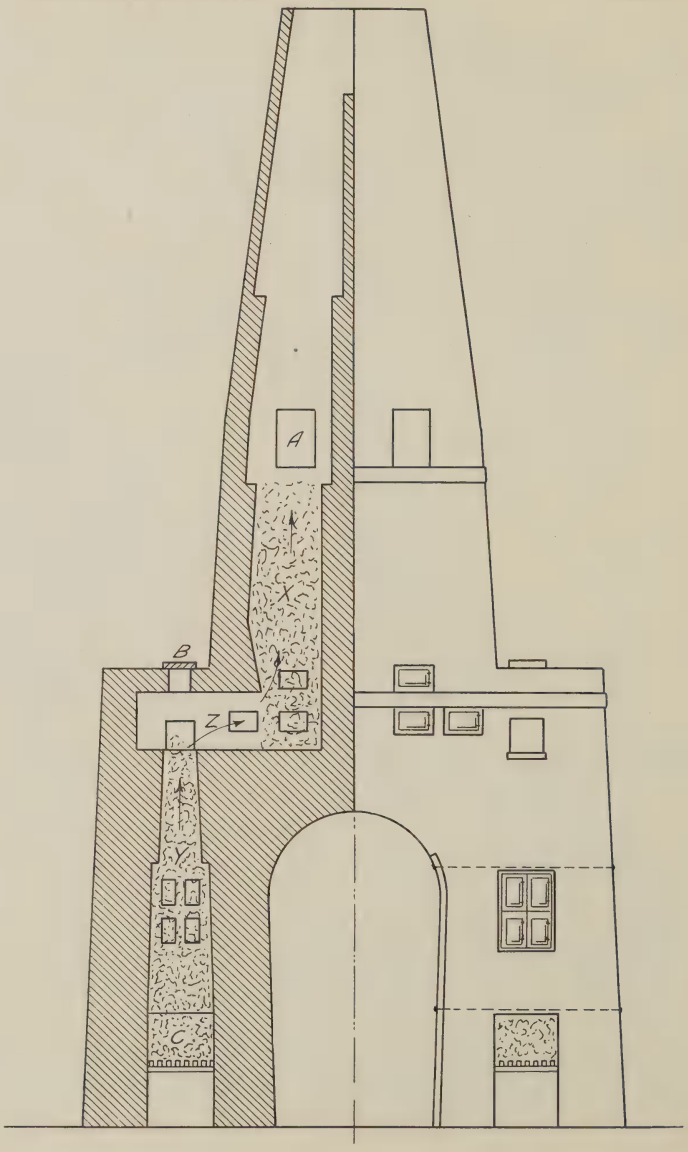
In parts of this country, it has become a feature of the regular business of some factories to produce what are called 'dobies.' These dobies or blocks are about 4 in. by 10 in. by 10 in. and are made usually on an auger machine from thoroughly weathered plastic fire clay, which has been run thru a dry pan having screen slots varying from ⅛in. to 5/16 in. wide. They are usually burned in round downdraft kilns from 28 feet to 36 feet in diameter, altho they may be burned between the bags in a kiln of regular ware.

In one section in the Middle West, where the clay is very plastic, difficulty is experienced in thoroughly oxidizing these dobies, and for this reason two oxidizing temperatures are made use of; the first one at 1100° F., held for 3 to 5 days, and the second at 1500° F., held 2 to 4 days, followed by the finishing temperature, held 20 to 40 hours, making the total time required to burn, 10 to 15 days. The temperature variations in the kiln are from cone 6 near the kiln wall, head-high, to cone 4 in the center of the kiln near the floor. The coal consumption is from 28 to 80 pounds per 100 pounds of burned dobies. The selling price of the dobies is $3.50 to $4.50 per ton.

The dobies are used as a non-plastic or grog and must, of course, be reground. The question naturally arises, cannot a fireclay be calcined just as it comes from the pit or mine, without going through a preliminary process of grinding and forming? Also, cannot some continuous process of burning be devised, whereby greater fuel economy may be had?

Another method, but without the grinding and forming, is being pursued in the calcining of flint clays at a number of factories in Ohio, Pennsylvania and Kentucky, where down-draft periodic kilns are used. The larger pieces of clay are selected for calcining, and the setting is carefully done, especially around the floor openings, so as to leave ample draft spaces. While a satisfactory quality of calcine may be secured by this method, and the preliminary grinding and forming is eliminated, yet it is far from economical. Care has to be taken in the setting or filling of the kiln; emptying is laborious, and it is quite a task to clean the kiln bottom after the burn. Furthermore, the operation of burning is periodic, and therefore loss of fuel is considerable. The

TRANS. AM. CER. SOC. VOL. XVIII FIG. 2 HARROP



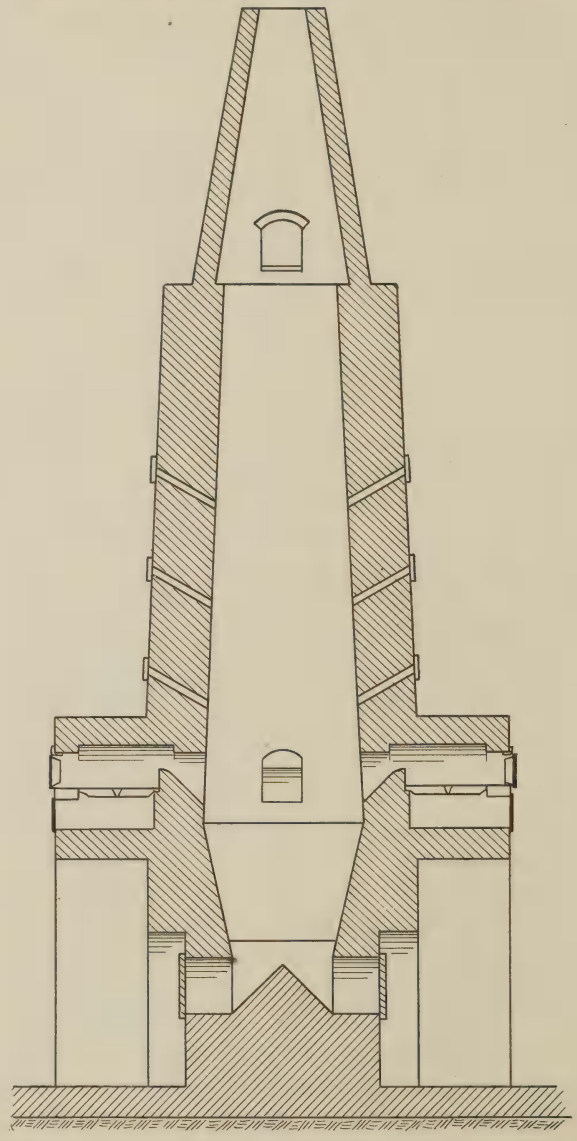
cost of calcining by this method is said to be $1.50 to $1.60 per ton of calcine. If the raw clay costs $1.00 per ton, mined, this would give a total cost of production of $2.60 to $2.75 per ton of calcine.

An analysis, from the physical structure standpoint of a clay calcined before grinding and of one calcined after grinding, will show some differences. If the clay is calcined before grinding, it will "crackle" to a greater or less degree, shrinkage cracks will show up in all directions and structurally, so far as the original lump is concerned, it is ready to fall to pieces. The small layers or plates and slivers, which remain intact, are very hard and brittle, and crushing, which is easily accomplished, results in the production of sound, angular, sharp-cornered fragments, which afford an excellent surface for the adhering of the bond clay.

On the other hand, when grinding the raw clay, the tendency is to produce rounded fragments, which are still further rounded and worn away in passing through the tempering mill and the auger machine. Upon crushing the burned grog, we find the grains to consist of a number of more or less rounded grains of smaller size, cemented or bonded together to a questionable degree and comparatively unsound.

At various places over the country are seen vertical shaft kilns. Some were intended to operate continuously, while others are merely up-draft periodic kilns. In the St. Louis district are some shaft kilns of this latter type. Figure 1 illustrates one of these kilns. It has an inside diameter of 15 feet and will hold about 50 tons of clay. It has one charging door at the level of the trestle, two drawing doors at the bottom, six grate bar furnaces, 24 in. wide by 30 in. long, and is burned off in about 10 days. In charging this kiln, the lower part is first lined with old railroad ties or other heavy timbers, the lower end resting on the kiln floor near the center and the upper ends resting against the wall above the furnaces. The clay is then charged in from above, filling the remaining interior. In this way the furnaces have a free space into which to send their flame and gases, although later they are probably more or less choked by the clay when it drops to the floor after the timbers are consumed. No temperature

TRANS AM. CER SOC VOL XVIII FIG. 3 HARROP



measurements are taken, the aim being to secure as high a temperature as possible.

In view of the increasing need of conservation of our fuel supply and reduction of manufacturing costs, the continuously operated kiln with its preheating and regenerative features is bound to come into more general use. If the saving is welcome to the manufactureer of clay wares, it should be equally welcome in the preliminary heat treatment or calcining of clays.

The vertical kiln used in our modern lime plants is a fairly economical device. The stone is preheated by the escaping combustion gases, but the heat from the burned lime is not saved as it might be, — cold air being used for combustion.

In the metallurgical industries, we find wonderfully devised apparatus for the purpose of roasting ores, etc. These furnaces are very economical of fuel, but for a temperature of cone 10 or thereabouts they would be mechanically unfit.

For the calcining of clays, we know the Europeans use two types of continuously operated vertical kilns — the Etagenofen or stage kiln and the Schachtofen or shaft kiln.

In the earlier efforts at Portland cement manufacture in this country, the Dietzsch kiln was used. This was of the Etagenofen type and usually consisted of two vertical shafts with offsets or shelves midway between the charging opening, which was near the top and the drawing opening near the bottom. Figure 2 shows this type of kiln, part in section and part in elevation. The raw material is charged in through opening **A** and fills the upper leg of the shaft **X**. Fuel is fed into the opening **B** on top of the hot material in the lower leg **Y**, space **Z** serving as a combustion chamber. As the material at the bottom of the lower leg of the shaft cools, it is drawn through opening **C**. The material at the bottom of the upper leg is then worked with an iron rod over into the space in the top of the lower leg. Fuel is dropped in at **B** and another charge of material put in at **A**. The air for combustion in passing up through the lower leg becomes very hot by the time the combustion chamber is reached and the combustion gases rising through the upper leg, preheat the newly charged material. Thus we have regenerative and preheating benefits and great fuel economy. The disadvantage of such a kiln, how-

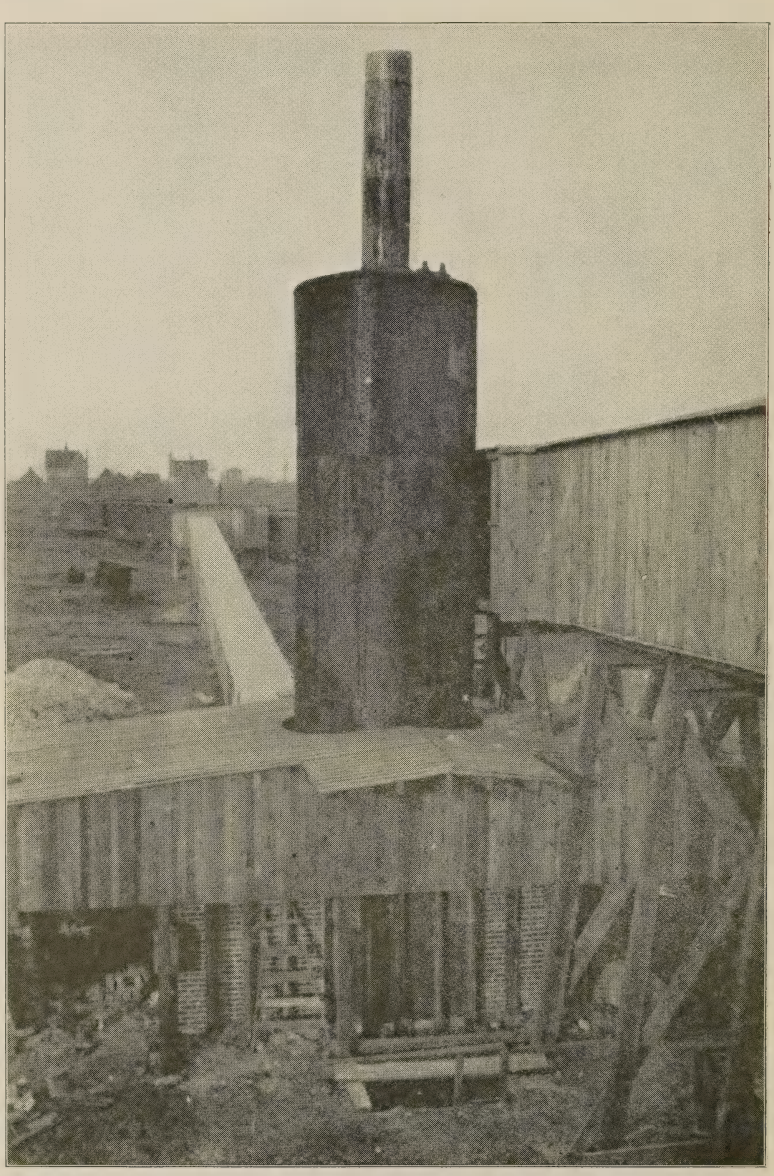


Fig. 4.

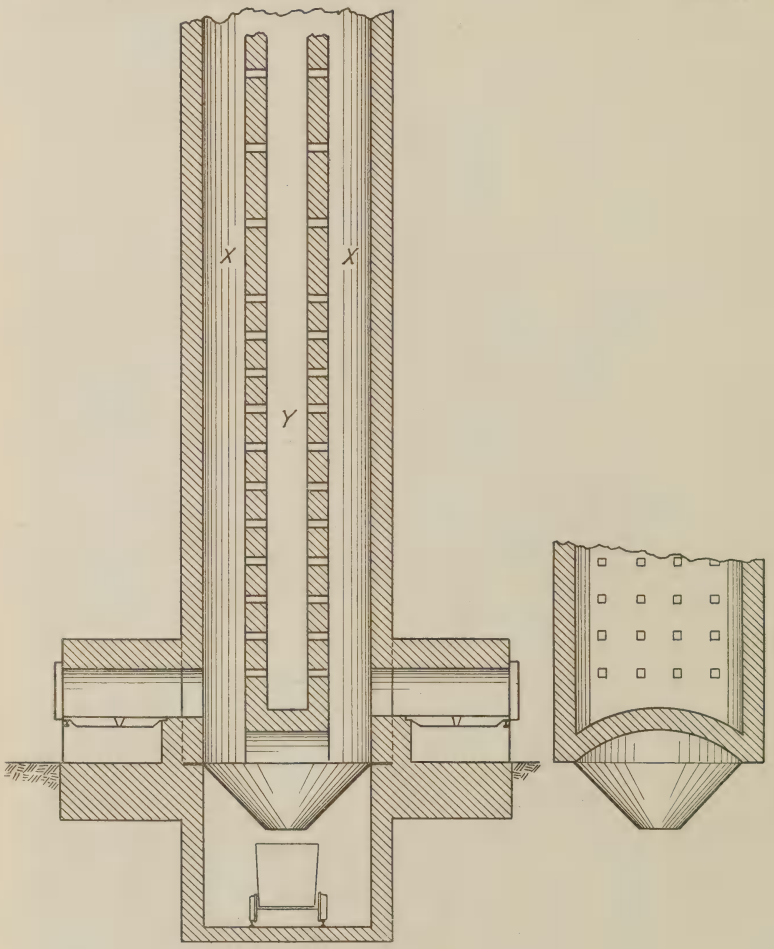
ever, lies in the hand labor of working all of the material across the bottom of the combustion chamber to the top of the lower leg, where the temperature is intense.

In the vertical shaft kiln, Figure 3, with proper shaping of the interior (larger toward the furnaces) and with a material, which does not become sticky upon heating, there should be no difficulty in the natural progress of the charge from the top to the drawing doors. It would seem, therefore, that this kiln, burned with producer gas or with attached furnaces operated as producers, and having all the economy features of the Etagenofen, should be the kiln best adapted to the calcining of clays. It is believed that this notion is prevalent with a great many engineers, who have had no actual experience with such a method of calcining. The writer has advocated such a kiln, but upon entering into a recent investigation of present-day methods of calcining clay in this country, was much surprised in being able to locate only one firm calcining clay in a vertical shaft kiln operated continuously; and the statement from this firm was to the effect that they did not consider the method satisfactory and intended discontinuing it.

The great barrier to the use of this type of kiln is the difficulty of maintaining a draft by natural means through the shaft of clay, and although it has been tried in a number of different places, it has been given up as impracticable. The desired temperature in some cases could be reached in time, but the daily capacity was so small and the annoyance and uncertainty of results so great that less economical methods were reverted to in order to secure the desired output.

One of the most recent attempts to calcine plastic clay in a kiln of this kind with natural draft was made in the kiln shown in Figure 4. The steel shell is an old oil tank with a steel stack riveted to the top and a ½ in. steel plate discharge cone with sliding gates riveted to the bottom. The shell is, of course, lined with fire brick, the inside diameter being about 5 feet. There are four furnaces, each about 20 inches wide by 5 feet long. Several attempts at calcining were made with this kiln, but each was a failure. While the clay did not slake or crackle badly, yet the fire would not penetrate the charge more than 18 inches toward

TRANS. AM. CER. SOC. VOL. XVIII FIG. 5 HARROP



the center and not more than 24 inches above the furnace throat. As the kiln was charged with clay from above, the draft became weaker and further progress ceased.

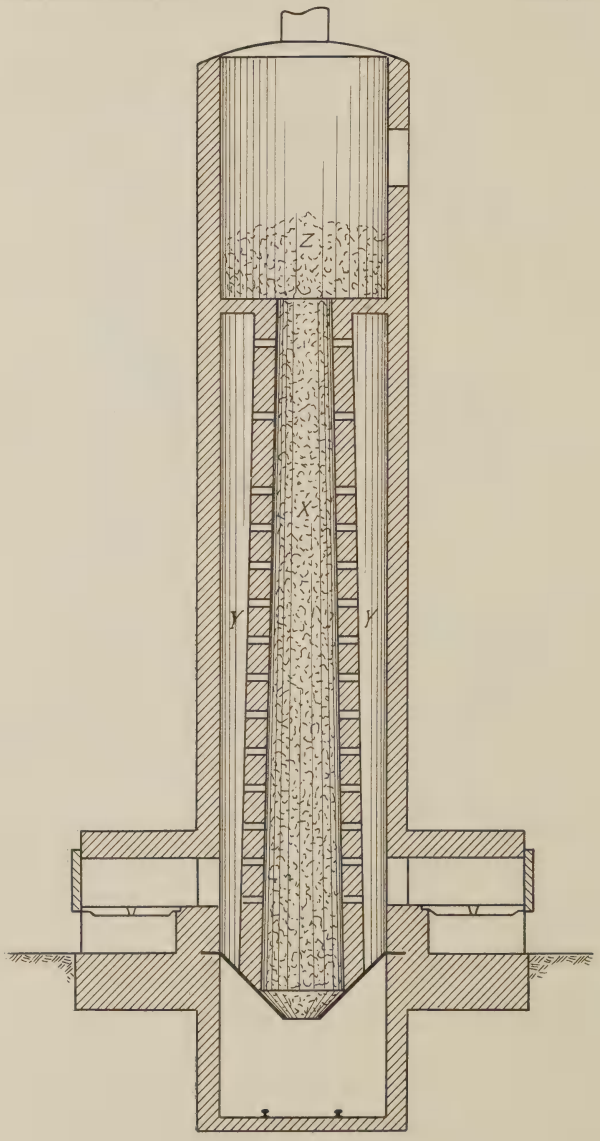
The scheme shown in Figure 5 was next resorted to. **A** and **B** are two perforated walls, resting on the arch **C**. Clay was charged down the spaces **X** and **X**. Space **Y**, which is rectangular in plan, served as a chimney. The furnace gases can go either up through the clay or, if there is too much resistance, they can pass through the perforations into the chimney **Y**. So far as draft was concerned, success seemed sure, but when they came to draw the calcined clay from the narrow spaces **X** and **X**, they met defeat.

The owner now proposes to build a circular muffle within this kiln, as shown in Figure 6. The clay will be charged in at the top and will descend through the shaft **X**. The furnace gases will pass into the annular space **Y** and either rise to the top or pass through the perforations into the clay and then rise. Regardless of which course the gases take in passing upward, they must, before leaving the kiln, pass through the clay at **Z**, the thickness of which can be adjusted so as not to choke the draft too much, but at the same time, permit considerable heat to be extracted from the combustion gases passing through.

One of the oldest factories in the East is using two continuously operated shaft kilns for calcining flint clay of the Mercer Horizon. The kilns (Figure 7) are about 6 feet inside diameter, and the height from the floor to the bottom of the chimney is about 21 feet. With a maximum temperature of cone 7, it require about 3 days for the clay to pass through. The capacity of each kiln is 4 to 5 tons of calcine per 24 hours, and the coal consumption is 650 pounds per ton of calcine. One day-man and one night-man do all the work in connection with both kilns. If only the large lumps that come from the mine were used, a larger capacity could probably be secured, but as run-of-mine clay is used, there are not enough flue spaces to permit of rapid firing.

Choking of the draft spaces, due to the crackling and compacting of the clay charge, appears to be the great obstacle to the success of this type of kiln. There would be little question as to economy, if the firing could be carried on as rapidly as desired.

TRANS AM CER SOC VOL XVIII FIG. 6. HARROP



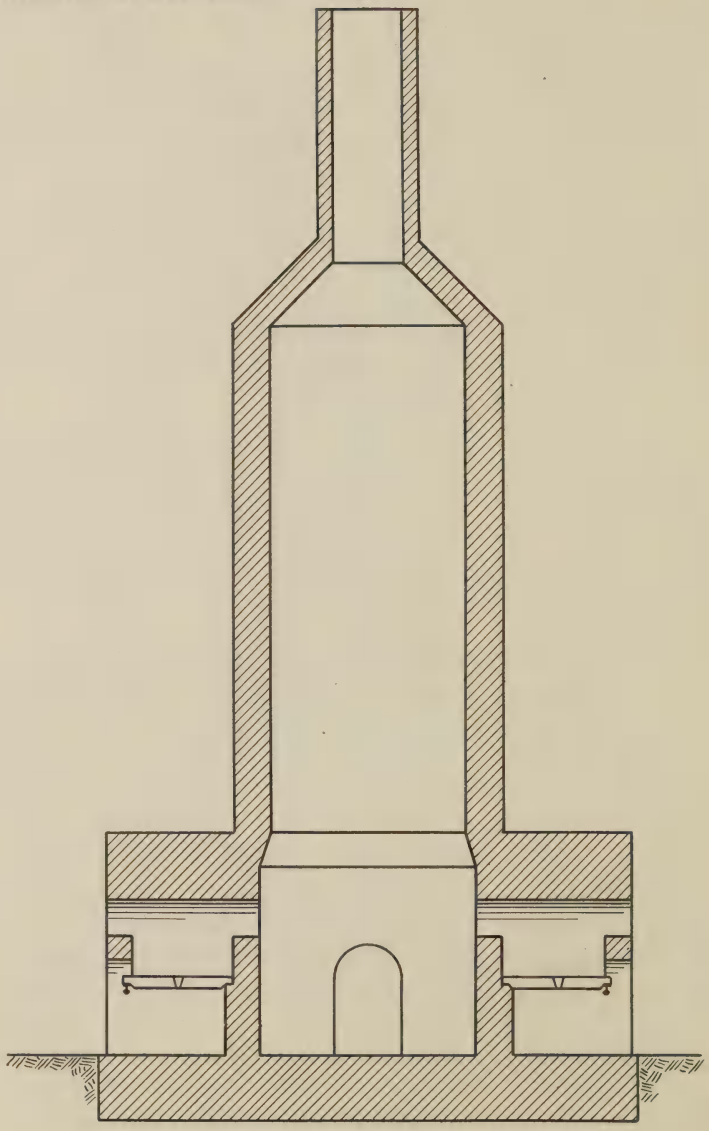
While rapid firing is practically impossible with natural draft, mechanical draft could be resorted to. The furnaces could be operated as pressure producers, with steam jets underneath the grate bars. A small centrifugal blower at the bottom could be used to blow air up through the calcined clay. When this highly heated air reaches the combustible gases from the furnaces, perfect combustion would result in close contact with the clay, — the hot products of combustion passing up would heat the clay above. As air forced in from below might produce a back pressure on the furnaces, it would be better if the air circulation or draft could be effected by an exhaust fan drawing from the top of the kiln. With this arrangement there would be no pressure to retard the flow of gases from the furnaces, although the cost of construction would be greater.

Even were we able to burn quickly in this continuously operated shaft kiln and do so with economy of fuel, there is one point that cannot be lost sight of. Practically all fire clays, whether plastic or flint, contain more or less carbonaceous matter, which must be removed by oxidation. This process of oxidation requires time, and the larger the lumps of clay, the longer will be the time required.

Assuming a vertical shaft kiln with an inside diameter of 6 feet and with the zone of high fire extending from the furnace level to a point 10 feet above, and supposing that the clay requires 48 hours for thorough oxidation, we could figure on a capacity of about 9 tons of calcine per 24 hours. If 72 hours is required for oxidation, the capacity would be only about 6 tons.

Calcining in the rotary cement kiln has been suggested as a feasible method, with the argument that the clay could be crushed to pass, say a 1 in. mesh screen, and thus require a much shorter time for oxidation. There would probably be no difficulty in securing a sufficiently high temperature, but the great cost of the equipment would probably not be justified under present conditions of demand for this material. A rotary kiln 150 feet long, erected complete with lining and chimney, but without feeding device or discharge conveying system, will cost about $15,000.

TRANS. AM. CER. SOC VOL. XVIII FIG. 7. HARROP



The writer had the opportunity recently to examine drawings covering a kiln for calcining plastic clays, in which the idea of the continuous chamber brick kiln was followed. There were two rows of comparatively small chambers. These chambers were to be charged through a hole in the crown, the clay falling onto the floor, which sloped toward the drawing door. Oil was to be used as fuel and this was arranged to drop into combustion compartments in the partition walls. The direction of the draft was to be horizontal, and the full continuous principle of burning was to be employed, with the attending fuel economy. In order to get the heat out of a mass of clay quickly, air must be permitted to circulate through it. If we are to heat up a mass of clay quickly, the combustion gases must be able to flow through it readily. Therefore, in order that this kiln operate economically, it would be essential that the air for combustion and the combustion gases pass through from six to eight chambers. The writer does not consider this practicable with ordinary pressures.

THE INFLUENCE OF VARIATIONS IN WATER ADDITION, LIME ADDITION AND TEMPERATURE IN THE SETTING OF PLASTER OF PARIS

BY C. H. KERR AND R. J. MONTGOMERY, TARENTUM, PA.

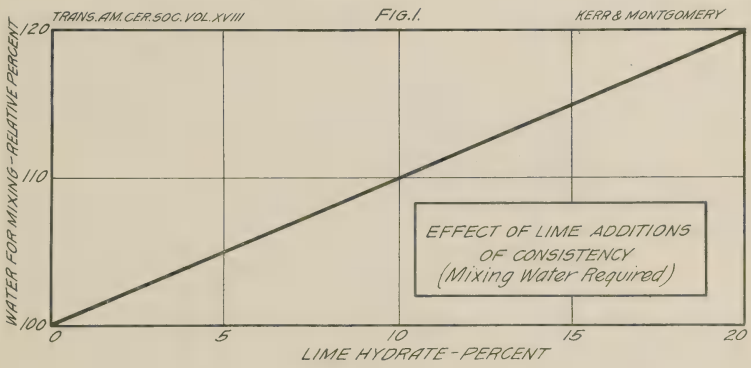
In the factory manipulation of plaster of Paris the best plaster may be rendered useless by improper handling, and conversely the most advantageous conditions must be determined and adhered to if the best results are to be obtained. It is a matter of common knowledge that, after setting has started, further agitation or disturbance of the plaster is very detrimental, if not fatal, to proper setting. Nevertheless, this is a very common cause of failure of plaster. The manufacturers of plaster are constantly being called upon to correct troubles with their products, when investigation shows the cause of the trouble to be agitation after setting has started. Certain other causes of variation that have received less attention are here discussed. There is very wide room for experimentation on many phases of variations in setting, due to variation in conditions surrounding the use of plaster.

In the present report only a summarized presentation of the data is made. The curves given cover a wide range of data on commercial plasters. varying in purity from approximately 100 percent gypsum in the original rock down to about 70 percent gypsum. It has been found that the same general effects are produced with the impure as with the pure plasters. These data have all been assembled and expressed as simply as possible, basing results as far as possible on percentage effects, rather than on absolute numerical data. The mass of figures from which these curves are derived would lead only to confusion if given in full, and the percentage change in results shows much more clearly the nature of the effect.

VARIATIONS IN LIME ADDITIONS

In many uses of plaster of Paris it is customary to add hydrated lime. In some cases freshly burned quick lime is slaked with water, and the slaked lime added to the plaster while still warm. It is more common, however, to add the commercially hydrated lime. The quantity added varies with the nature of the application, but is usually from 4 or 5 to 20 percent. In Figures 1, 2 and 3 are shown respectively the effects of variation in lime addition on:

Consistency — Figure 1.
Time of set — Figure 2.
Tensile strength — Figure 3.

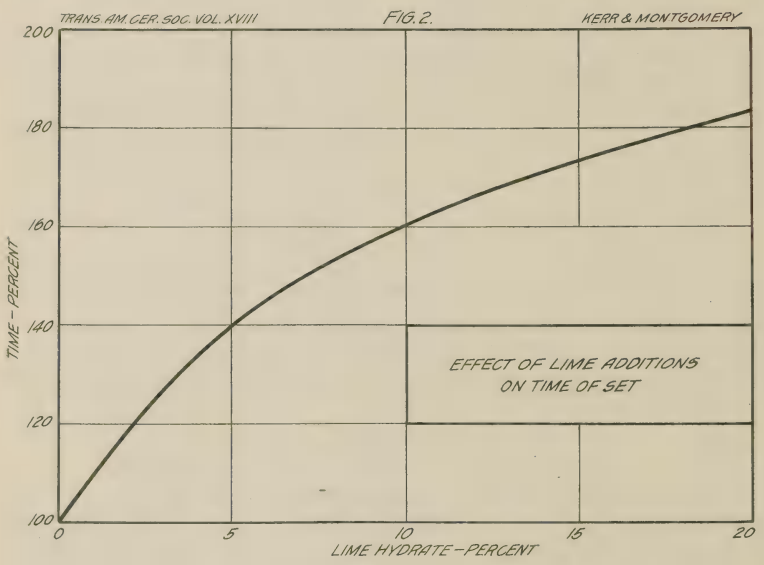


The data, as has been previously stated, are compilations of percentage effects on many different plasters and different limes. The nature of the effect with all the different kinds of plasters and limes has been found to be the same, the differences being only in the absolute values of the results.

In the limes, there has been found to be substantially no difference between practically pure calcium hydrated lime and magnesium limes with MgO up to 20 percent; no hydrated limes with over 20 percent MgO have been studied. So far as has been covered in this work, the physical results produced seem to be largely independent of the chemical composition of the lime.

Consistency-Lime Relation. The curve in Figure 1 shows that the amount of water that must be added to the plaster or

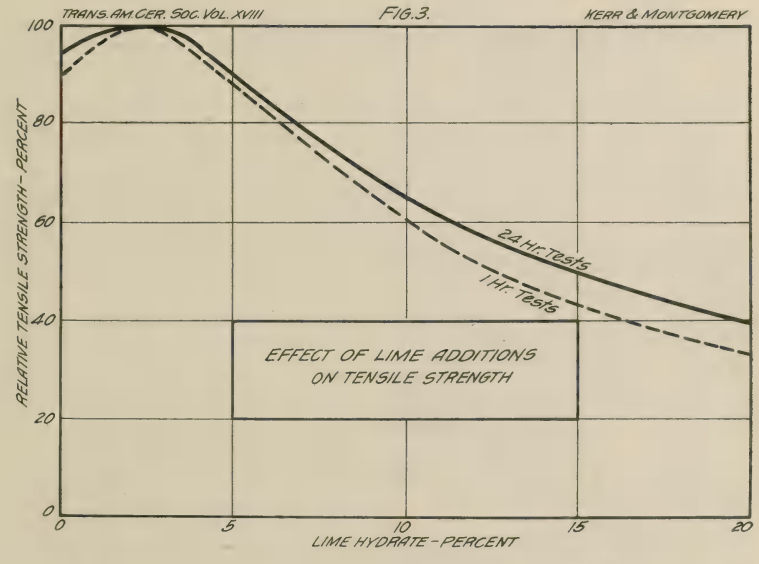
plaster-lime mixture, to give a standard consistency, increases in direct proportion to the amount of hydrated lime added. Each 1 percent lime addition increases the necessary amount of mixing water to give standard consistency by about 1 percent. The consistency figure referred to is determined by us as the number of cubic centimeters of water per 100 grams of plaster or plaster-lime mixture which will give a mixture just thin enough to flow 10 inches down a right-angled, copper-lined trough inclined at 30° from the horizontal. For a description of methods of testing



see paper elsewhere in this volume entitled "Factory Methods for the Testing of Plaster of Paris."

Time of Set-Lime Relation. Experimental results with different plasters and limes show the effect on the time of set of variations in the amount of lime hydrate added. These results expressed on a percentage basis are shown in Figure 2. The first additions increase the time of set proportionately more than further additions. With 5 percent lime, the time of set is increased almost 40 percent, with 10 percent lime a little over 60 percent and with 20 percent lime about 80 percent.

Tensile Strength-Lime Relation. In Figure 3 is shown the general effect of lime additions on the tensile strength after one and twenty-four hours' standing. A peculiar phenomenon is here observed. The first additions increase the strength to a maximum at about 2 to 4 percent lime hydrate content. Further additions decrease the tensile strength, the additional decrease being less with each subsequent addition. With 10 percent lime hydrate in the mixture, the strength is a little over 60 percent of maximum



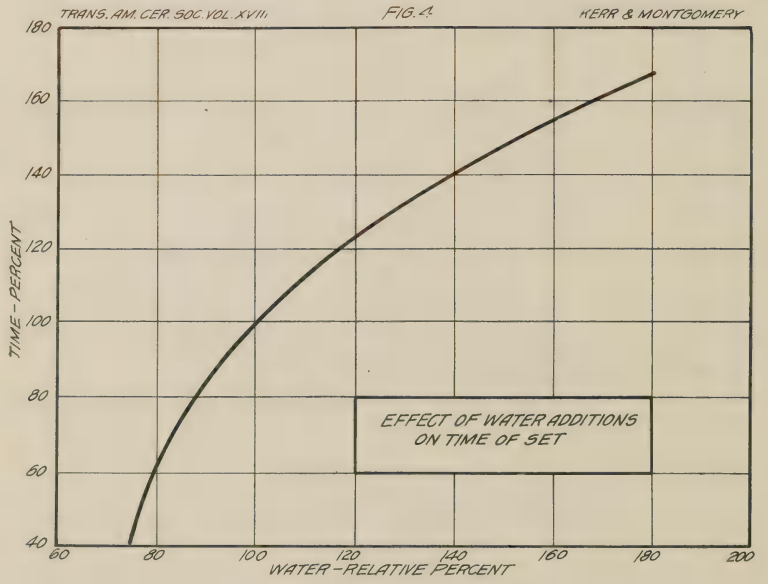
strength; with 20 percent lime hydrate, about 40 percent of the maximum strength.

Temperature Lime Relation. The retarding effect of lime hydrate additions is shown in Figure 2 — the time of set curve. In the temperature curve, or heat evolution curve the corresponding retarding effect is found, but the amount of heat evolved is exactly proportional to the amount of active plaster present, the lime exerting no effect beyond its action as a dilutent and as a retarder.

VARIATIONS IN WATER ADDITIONS

One of the very common causes of trouble in plaster application is the use of a wrong amount of water in approximately constant quantity, or a still more common phase of the trouble is a variation in the amount of water used. The importance of proper regulation of water content is seldom recognized.

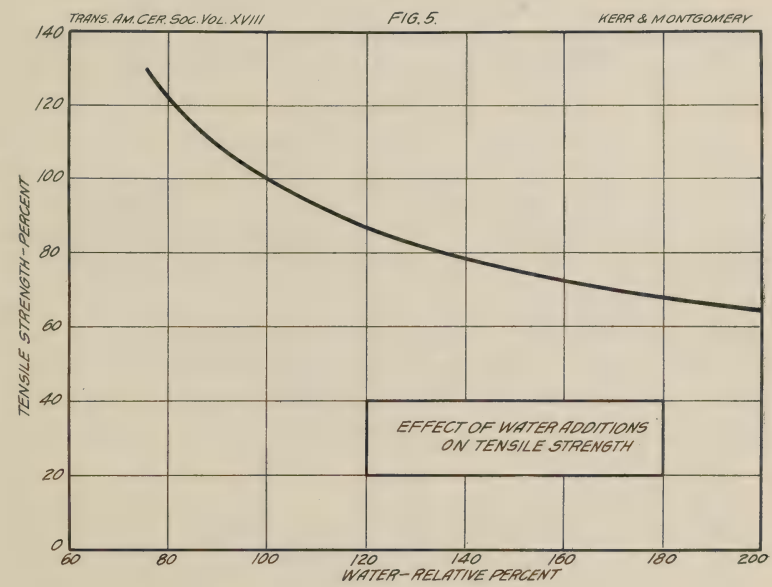
The curves shown in Figure 4 and 5 are composite curves expressed on a percentage basis, the data, as previously, being



taken from a very large number of tests on many different plasters of varying purity.

Time of Set-Water Relation. Figure 4 shows the enormous effect of water addition upon the time of set. The 100 percent water figure represents standard consistency; that is, the number of cubic centimeters of water which must be added to 100 grams of plaster to give a mixture just thin enough to run 10 inches down a right-angled, copper-lined trough, inclined at an angle of 30° from the horizontal. Very slight changes in the water

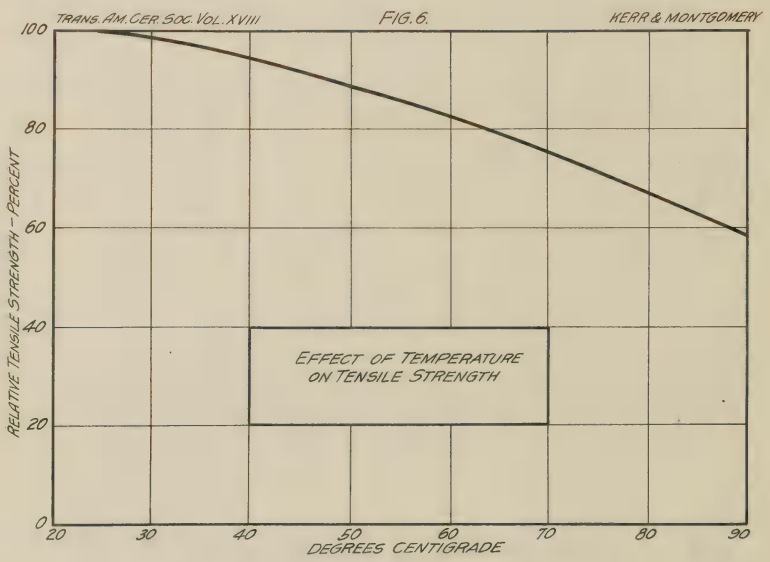
content at or below this standard consistency figure considerably affect the time of set. If the water is decreased to about 75 percent of the standard consistency (a very thick cream), the time of set is only about 40 percent of that required at standard consistency. This part of the curve varies considerably with different plasters, but in all cases the effects are tremendous. With water additions above standard consistency, each increase in water increases the time of set, the proportionate effect lessening with each subsequent adition of water, until at 200 percent or twice the



quantity of water required for standard consistency, the time of set has increased about 80 percent.

Tensile Strength-Water Relation. Basing these data on 100 percent strength with 100 percent representing the amount of water required for standard consistency, Figure 5 shows the decrease in strength with increase in water. From about 30 percent greater than normal strength with 75 percent of the consistency water, the strength decreases to about 65 percent of normal when the water is about twice that required for standard consistency.

Temperature Curve-Water Relation. In Figure 4 is shown the effect of water additions on the time of set. In taking temperature curves, this same retarding effect of increase in water is of course observed in the curve; beyond this retading effect, and a corresponding effect on the temperature increase of the increased quantity of water present, it is found that variations in water condition do not affect the temperature curve. The total heat evolved is the same, and if calculations are made of the heat required to raise the temperature of the particular quantity of



water used, it will be found that the total heat is independent of the water addition. This makes it possible to calculate evolution of heat to a standard basis regardless of the amount of water used.

VARIATIONS IN TEMPERATURE

In certain special applications when the temperature of the room or a special receptacle is not approximately atmospheric, it becomes essential to consider the effect of the surrounding temperature on the setting. The curve in Fig. 6 shows this relation on a tensile strength basis, this curve like the preceding ones

being a composite one including many determinations on different plasters. With increase in room temperature above 24° C., the tensile strength of the set plaster decreases with each increase in temperature until at 90° C. (the highest temperature here considered) the strength is reduced to about 40 percent.

SUMMARY

The addition of hydrated lime to plaster shows the following effects:

1. The amount of mixing water required to give standard consistency increases in direct proportion to the amount of lime hydrate added, each one percent increasing the amount of water by one percent.
2. The time of set is increased.
3. The tensile strength is increased somewhat with small additions to a maximum at about two to four percent lime hydrate but is greatly decreased with larger additions.

Variations in mixing water added showed the following effects.

1. The time of set is increased with each increase in water.
2. The strength is decreased with each increase in water.

The higher the temperature of the air surrounding the setting plaster, the lower is the strength of the resulting product.

METHODS FOR THE FACTORY TESTING OF PLASTER OF PARIS

BY R. J. MONTGOMERY AND C. H. KERR, TARENTUM, PA.

In this paper we shall deal only with the commercial testing of plaster of Paris shipments with the view of arriving at a decision as to the nature of the tests to be applied and the details of manipulation in the respective tests. No attempt is to be made to classify plasters or to correlate methods of manufacture with resulting physical properties.

In ordinary consideration of plaster the time of set and tensile strength are most commonly referred to, and in very many cases no other physical properties are considered. These two tests are usually of comparatively little value in testing commercial shipments (especially the tensile strength test), unless considered in connection with other data. Of the many kinds of tests that might be made, the temperature curve during setting is probably the most important and most promising.

Briefly the tests to be considered and the methods are as follows:

Chemical Analysis. The methods of analysis are the ordinary methods of analytical chemistry. Moisture is commonly determined at 80° C. and combined water at 220° C. The chemical composition is of little or no value in comparing various shipments of a given plaster. Determinations of water content are usually misleading, since they show variations in atmospheric conditions and in conditions of exposure more than variations in original materials or in methods of manufacture. Frequently the water determination is nothing more nor less than a determination of the age of the plaster.

Consistency. The amount of water that must be mixed with plaster to give a body of the correct working consistency is of considerable importance in judging the nature of a plaster. This may be determined as the amount of water (in cubic centi-

meters per 100 grams) that must be added to the plaster to give a mix that will just pour smoothly from a cup. But a better method is to determine the amount of water (in cubic centimeters per 100 grams) to be added to the plaster to give a mix of such viscosity that it will flow just 10 inches down a copper-lined through inclined at an angle of 30° from the horizontal. The consistency figure is of considerable value both in comparing different plasters and in checking the uniformity of a single product.

Start of Set. This has not often been determined though it is of great importance. It is an empirical and not an absolute point, but it is satisfactory, in commercial testing, to consider the start of set as that point at which the mixed plaster has just begun to be rigid, and a convenient method of determining it is to take the time at which the mix will just support an ordinary 12 inch chemical thermometer inclined at an angle of about 15° from the vertical. This start of set test is convenient when made on the same sample as the temperature curve. Two hundred grams of plaster and 150 cubic centimeters of water are mixed vigorously with a spatula for one minute and then poured into a paraffined paper cup. The thermometer is immersed to a constant depth of 1¾ inches and held in the fingers inclined at 15° from the vertical. A little skill is necessary in making the test, but it soon becomes possible with very few trials to judge when the setting is just sufficient to support the thermometer and prevent its tipping over. The test is of prime importance since it is a well established fact that plaster is easily "killed" by continuing the mixing or working after setting has started.

Time of Set. The time of set has been determined by thumb nail indentation, lifting from a glass surface, etc., but the best method is some modification of the penetration needle, and a very satisfactory test is to determine the time which must elapse during the setting before the plaster is sufficiently hard to bear the weight of 2½ pounds on a needle 1/16 inch square at the point without breaking the surface further than to leave a clear impression of the square point. In making this test, 100 grams of plaster and an amount of water corresponding to the consistency figure determined as described previously, are

mixed vigorously for one minute and poured into any convenient mold to form a slab about ¾ to 1 inch thick. The top is then struck off quickly with a spatula. While this time of set test is of undoubted importance in many applications of plaster as showing the time range for proper working, the test must not be considered the most important one, and too much faith must not be placed in conclusions drawn from uniformity or lack of uniformity in time of set.

Temperature Curve. The method of making a temperature curve is one that invites much discussion. One satisfactory method is to mix 200 grams of plaster with 150 cubic centimeters of water and read the temperature changes by a thermometer immersed in the mix. The plaster and water are mixed vigorously for one minute and quickly poured into a paraffined paper cup, a cup being discarded with each test, thus saving the cleaning up of dishes. A regular 12 inch chemical thermometer is immersed 1¾ inches and held in the fingers inclined at an angle of about 15° degrees from the vertical. Start of set is noted as described previously. From time of pouring of the plaster mix, one minute more is allowed (making two minutes from the time the mixing started) before the first temperature reading is made, and this is noted as the two minute point on the temperature curve. Temperature readings are then made every minute until the maximum temperature has passed. The original temperature is taken as the average of the temperature of water and plaster, both being kept in the working room some hours before testing, and the difference in temperature should never exceed 2° C. Of all the many tests that have been suggested and tried in plaster testing, the temperature curve determination is the most important and the one that offers the greatest promise of future usefulness. It is not unlikely that, with some further developments in our testing methods, it will be found quite satisfactory in many applications of plaster to confine the testing to the temperature curve only, reading from this curve the various data regarding rate and time of setting and judging from this curve also the character of the plaster as regards comparative strength and general properties. The temperature curve is of the utmost value in determining the regularity or uniformity of current shipments.

Tensile Strength. As in cement work, the tensile strength is determined on figure-8-shaped specimens after 1, 7 and 28 days and occasionally after periods of a few hours only. The tensile strength tests are usually of little or no value and in most testing work they may be omitted without loss.

Modulus of Rupture. Transverse breaks give more reliable and better data than tensile strength tests, and when strength tests are desired this is to be preferred. In most applications all strength tests may safely be omitted, because other tests give much more accurate and reliable clues as to the nature of any variations in plaster.

Sieve Test. Dry sieve tests should be made to show the residue on 100-mesh for in practically all uses the fineness is important.

Specific Gravity. Determinations of specific gravity have sometimes been recommended, but this has not been found to disclose information of any great value in the testing of plasters.

Circumstances must govern the choice of tests but for regular commercial testing of plaster shipments the tests to be selected will usually be,

Temperature curve during settting,
Start of set,
Time of set,
Consistency,
Sieve test,

with the possibility frequently of narrowing this down to the temperature curve and sieve test only.

METHODS OF MAKING THERMAL CONDUCTIVITY TESTS, AND THE TRANSMISSION OF HEAT THROUGH BRICKS

BY ROY A. HORNING.

There are several methods of making heat transmission tests on brick or any material, but owing to our limited time it will be necessary to only discuss the most important ones. Nearly all methods of determining the thermal conductivity or heat transmission are based upon modifications of two methods the hot air box method and the hot plate method. We shall take up first the hot air box method.

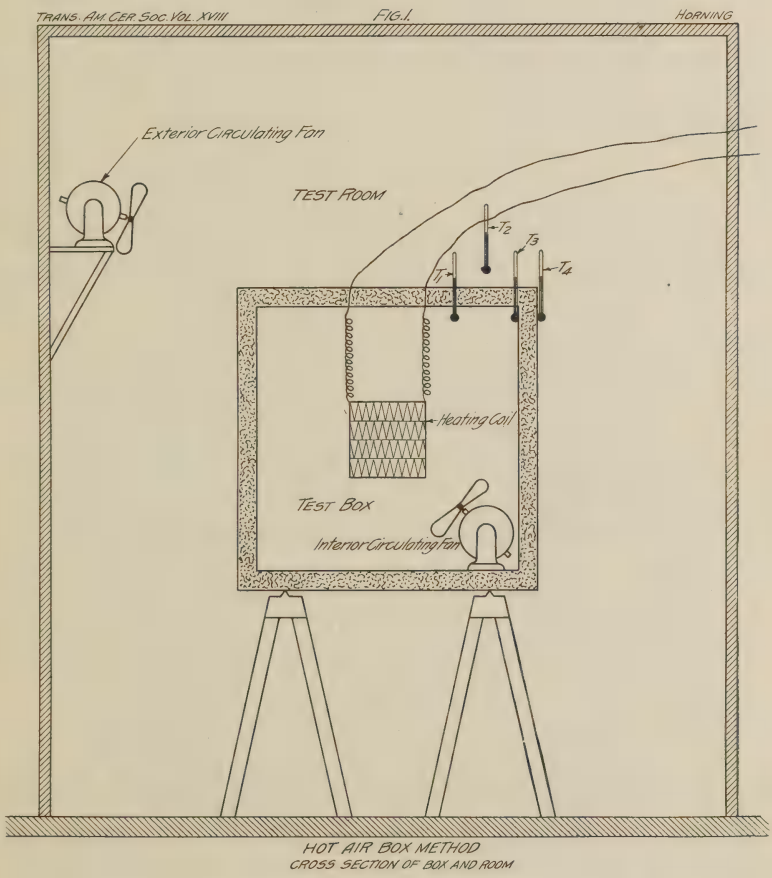
Hot Air Box Method. This method consists of building a box three or four feet square and as deep, and enclosing in it a heating coil and a fan to circulate the heated air. See Figure 1. In the interior of the box are placed two thermometer bulbs, one placed one inch away from the surface, and the other placed on the inside surface. Two are placed on the exterior of the box, one on the outside surface and one in the outside room, one inch away from the surface. These thermometers are arranged so that they may be read without entering the test room. This is accomplished by means of telescopes. The box is built on supports so that the air may circulate on all sides, also in the test room is placed a fan to promote outside circulation.

After making proper electrical connections, the current supplying the fans and coil is turned on, and 48 hours allowed to elapse so as to bring conditions to an equilibrium, thereby insuring uniform transmission of heat through the box on all sides. When conditions become constant, readings are taken every 10 minutes over a period of two or three hours.

The following data are taken:

The amperage of coil and fan.
The voltage of coil and fan.

The inside air temperature.
The outside air temperature.
The inside surface temperature.
The outside surface temperature.



At the end of two or three hours, as the case may be, the average of the foregoing data is computed and from this data is calculated the transmission in B. T. U.'s, per square foot of area, per ° F. difference in temperature per 24 hours. The following formula is used.

1 volt $\times$ 1 amp. = 1 watt.

1 watt flowing one hour = 3.416 B. T. U.'s.

Letting FA = amperage of fan,

FV = voltage of fan,

CA = amperage of coil,

CV = voltage of coil,

we have the watts supplied per hour:

$$(FA \times FV) + (CA \times CV),$$

or in B. T. U.'s we have

$$[(FA \times FV) \times (CA \times CV)]\ 3.416.$$

Letting D = difference in temperature,

letting A = mean area in square feet, then

the heat transmitted per 24 hours per square foot, per ° F. difference in temperature

$$= \frac{(FA \times FV) + (CA \times CV)\ 3.416 \times 24}{D \times A}$$

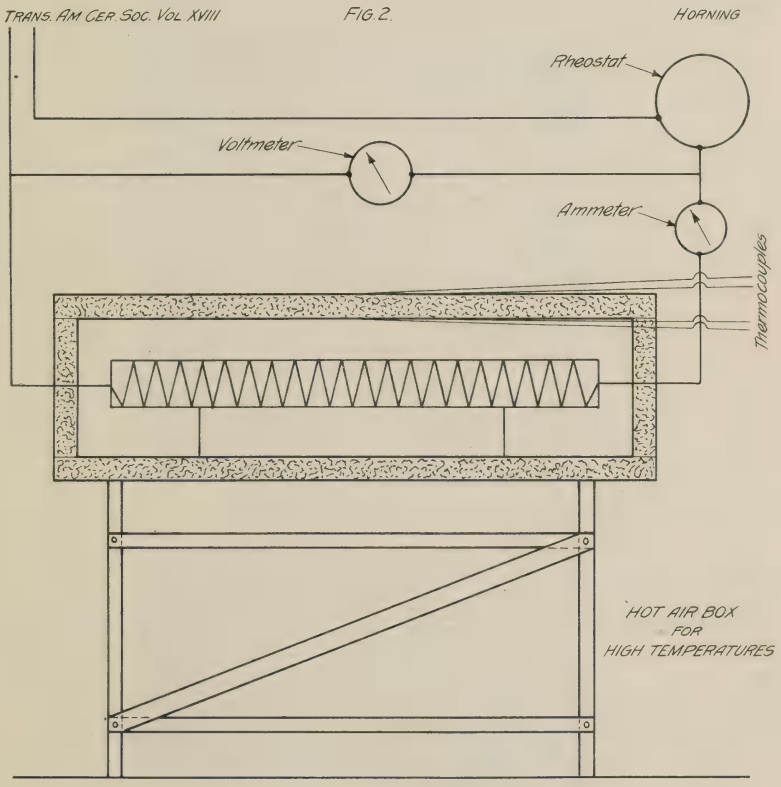
This transmission thus calculated can be reduced to the standard thickness of one inch by simply multiplying by the average thickness, since the amount of heat that will be transmitted is inversely proportional to the thickness, the material being homogeneous.

The transmission may be calculated on either the difference in temperature of the surface or the difference in temperature of the air. This should be specified.

The method just described must be modified for use at temperatures above 300° F. For temperatures exceeding 300° F., better results can be obtained by using a cylinder 8 to 10 inches in diameter and about 24 inches long. See Figure 2. Inside this cylinder is placed an electric coil as shown. The same method of running the test is used here as in the formerly described hot air box test excepting, of course, that there is no fan used. Thermocouples are used instead of thermometers.

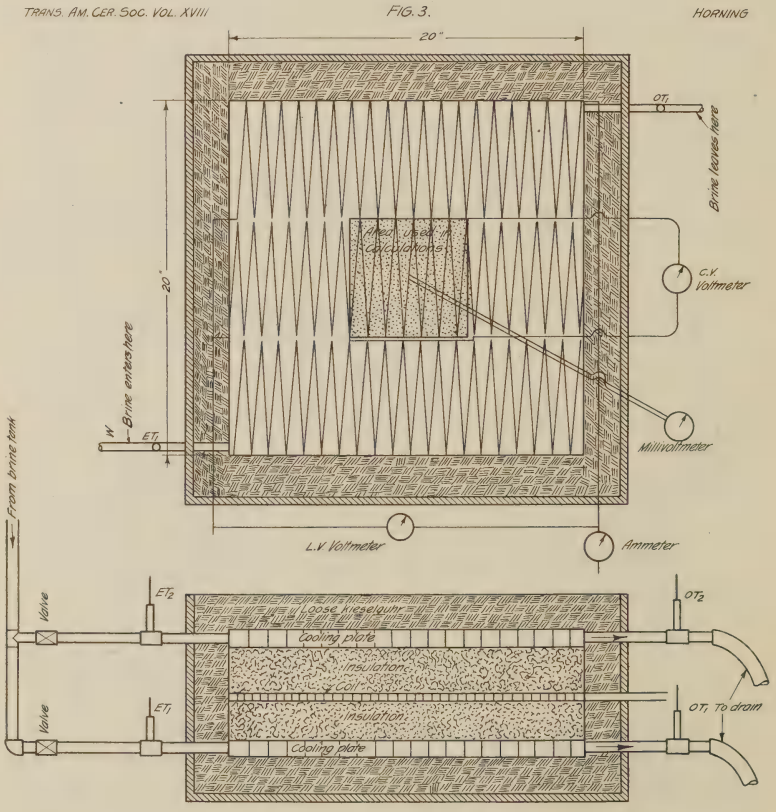
Hot Plate Method. Upon a table at least 30 inches square is placed a layer of loose asbestos or kieselguhr two inches thick.

Upon this is bedded a water plate 20 in. by 20 in. by 1 in., made of copper and so constructed that the water enters one corner and is made to travel back and forth across the face of the plate, leaving at the corner diagonally from the one through which it entered. This pan is leveled up, and upon it is placed a layer of



the material to be tested. A layer of three electric coils is placed upon this. The electric coils are 6½ in. by 20 in. made from No. 26 ⅜-inch nichrome ribbon. Nichrome leads are brazed onto the center coil at points which will give the voltage drop through the center one-third of its length. The object of this will be explained later. The three coils are connected in parallel and are kept

apart by means of 2 in. strips of asbestos paper. The junction of a thermo-couple is placed in the center of the 20 in. by 20 in. area and this is insulated by means of fine silica glass tubing. A second layer of the material to be tested, the same thickness as the first layer, is placed upon the coils, and upon this is laid a



water plate, a duplicate of the first. The whole apparatus is surrounded by a box at least 8 in. larger than the apparatus. This is filled with loose asbestos or kieselguhr, and a 2 in. layer of the loose material is placed upon the top of the upper water pan. This loose insulation protects the apparatus from any atmospheric changes, and also prevents losses by radiation. See Fig. 3.

A rheostat is placed in series with the coils, and an ammeter is inserted between them. A voltmeter is connected by means of a double throw switch so as to read either the voltage drop across the three coils, or across the one-third of the center coil.

The reason for measuring the voltage drop across the center one-third of the center coil is to find out how much heat is spent on the center one-ninth of the total area. One law of heat is "heat flows from the hotter to the colder body." It can readily be seen that the heat generated by the electric coils will flow in all directions, especially that generated on the outside edge of the coils. There is no way of knowing the amount of heat lost through lateral flow, but if the voltage and amperage of this small area in the center is known, it can be safely assumed that all the heat generated there by this amperage and voltage flows directly through the material being tested, since the temperature of the center one-ninth and the adjacent material is practically the same.

The following data are taken:

1. The temperature of the water as it enters plate.
2. The temperature of the water as it leaves plate.

The average of 1 and 2 is taken as the outside temperature at the center of the 20 in. by 20 in. layer of the material under test.

3. Coil temperature (by means of thermo couple, this is taken as the inside surface temperature).
4. Drop in voltage across center one-third of center coil.
5. Amperage of center coil $= \frac{\text{(Total amperage)}}{3}$

Forty-eight hours are allowed to elapse between turning on the electric current and the recording of data. The period of observations is every five minutes and lasts for two hours. The average of the twenty-four readings respectively of the foregoing data are used in the calculations.

METHOD OF CALCULATION

1 volt × 1 ampere = 1 watt,

1 watt = 3.416 B. T. U.'s per hour = 81.984 per 24 hours.

Heat put in during 24 hours = volt × ampere × 24 × 3.416.

Data taken. (See Figure 3.)

ET_1 and ET_2 = temperature of entering water,

OT_1 and OT_2 = temperature of leaving water,

LV = voltage drop through three coils,

CV = voltage drop through 1/3 of center coil,

CT = temperature of center coil by millivoltmeter,

Amperage of 3 coils.

Average of ET_1, ET_2, and OT_1, OT_2 = temperature on cold side of insulation,

CT = temperature on hot side.

Difference between CT and average of ET and OT gives difference in temperature used in calculation.

Heat transmitted in B. T. U.'s per 24 hours, per square foot area, per inch thickness, per ° F. difference in temperature

$$= \frac{\text{voltage} \times \text{amperage} \times 81.894 \times \text{thickness in inches}}{\text{° F. diff. in temp. between two sides} \times \text{area in sq. ft.}}$$

As an example:

500° F. = diff. in temp. between hot and cold side of material,

3 in. = thickness,

.617 = area in sq. ft.

15 = average voltage,

10 = average amperage, use 10/3 as average amperage of center coil, since coils are in parallel.

$$\frac{15 \times 10 \times 81.984 \times 3}{500 \times 0.617 \times 3} = 39.88$$

B. T. U.'s transmitted in 24 hours per square foot of surface per inch of thickness per ° F. difference in temperature.

The curves in Figure 4 show the amount of heat that will pass through a square foot of material one inch thick, per degree difference in temperature calculated on difference in temperature

from 250° F. to 1500° F. Curve No. 1 is of a vitreous brick with 5 percent absorption. Curve No. 2 is that of a high grade fire brick made from coarse material, and whose absorption is 10 percent. No. 3 is of a brick of pure diatomaceous earth. No. 4 is of a brick made from diatomaceous earth, clay and cork, the cork having been burned out in the process of manufacture.

Theoretically, bodies of a high specific gravity conduct heat more perfectly than bodies of a low specific gravity, so it must be concluded from this theory that if the object is to keep heat in, it is necessary to utilize bodies of low specific gravity.

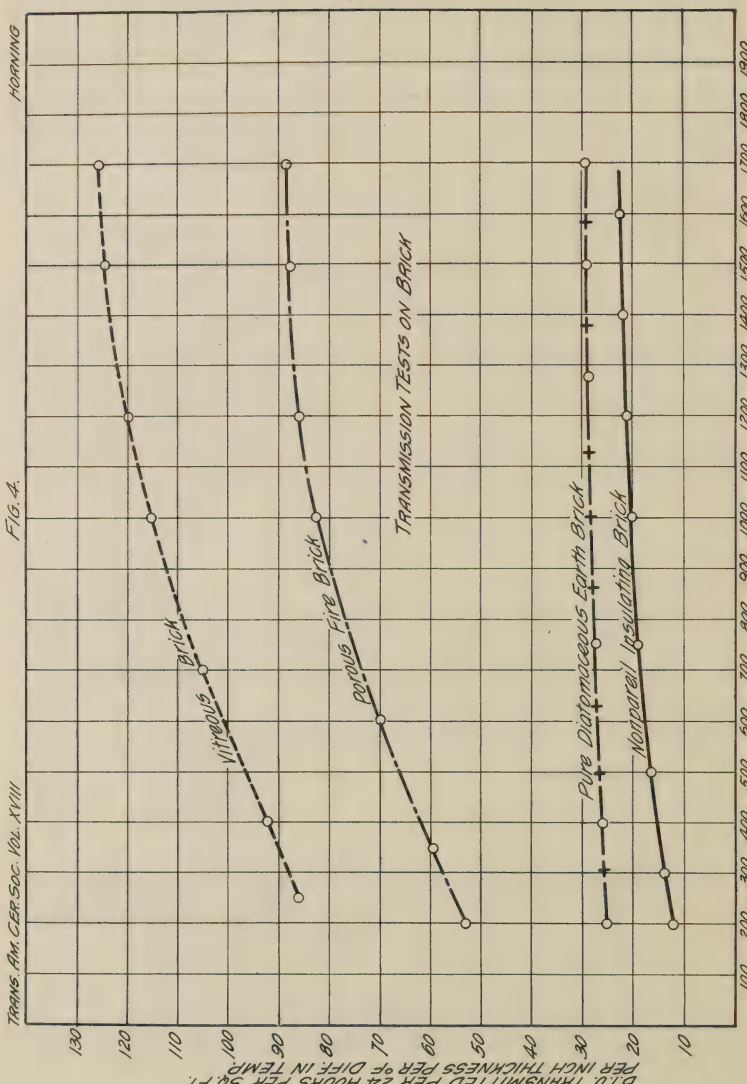
Air is conceded to be a poor conductor of heat, but even at very low temperature convection begins, and air loses its ability to retard heat flow. On the other hand, however, if the air can be prevented from circulating, it remains an excellent non-conductor even at very high temperatures. It is with this fundamental principle in mind that insulation problems are solved.

In the case of a vitrified brick we have a very dense body, each particle being in close contact with others, and in this case the heat travels through the brick by internal conduction. The porous brick becomes a poorer conductor, due to the fact that the contact between particles is poor, allowing the surface resistance to come into action, in addition to the air contained in the minute crevices which can not move.

The limits to which it is possible to make a porous brick leaves a good conductor of heat, when compared with brick made from materials containing confined air throughout its mass, as does diatomaceous earth.

Diatomaceous earth consists of the skeletons of prehistoric diatoms, which, according to geologists, existed in the Cretaceous period. Chemically these diatom skeletons consist almost wholly of pure silica, and physically they consist of many microscopic air cells, each cell being a heat retarding unit. The use of this material, and this is true of all loose materials, is quite limited, however, if it is formed into brick or other compact convenient shapes, its field as a heat insulator is unlimited.

Brick made from pure diatomaceous earth are very good insulators, but their structural strength is very low. In order to


TRANS. AM. CER. SOC. VOL. XVIII
FIG. 4.
HORNING
TRANSMISSION TESTS ON BRICK
Vitreous Brick
Porous Fire Brick
Pure Diatomaceous Earth Brick
Nonpareil Insulating Brick
B.t.u. TRANSMITTED PER 24 HOURS PER SQ. FT. PER INCH THICKNESS PER °F DIFF. IN TEMP.
DEGREES DIFFERENCE IN TEMPERATURE
10 20 30 40 50 60 70 80 90 100 110 120 130
100 200 300 400 500 600 700 800 900 1000 1100 1200 1300 1400 1500 1600 1700 1800 1900

give it this necessary strength, clay must be used as a binder. According to theory, however, this would lower its efficiency as a heat insulator, but, by experiment, it has been proven that it is quite practical to add, not only clay, but some combustible material such as cork that will be burned out in the process of manufacture, thus giving the brick, not only structural strength, but in addition insulating qualities that far exceed that of pure diatomaceous earth. The physical strength obtained in this manner is sufficient to permit its use as an integral part of the construction.

Referring again to curves in Figure 4, it can readily be seen that the conductivity per degree Fahrenheit difference in temperature increases as the difference in temperature increases. Taking curve No. 1, the conductivity per degree Fahrenheit difference in temperature at 200° F. is but 74 percent that at 1000° F. difference in temperature, and 68 percent that at 1500° F. difference in temperature. A similar change can be noticed in all curves shown. This change in conductivity is due to the change of the other physical properties of the body under the influence of heat.

Using the curves in Figure 4, to find the quantity of heat in B. T. U.'s that will pass through a wall of known thickness over a certain area, with a certain difference in temperature between the two sides over a period of twenty-four hours, follow the line of temperatures on the lower edge to the difference in temperature that exists in this particular case. Where the perpendicular passing through this point intersects the conductivity curve for the material in the wall, multiply the corresponding figure on the left hand side of the figure by the difference in temperature existing, multiply also by the area, and divide the product by the thickness of the wall in inches.

Example:

Wall of vitreous brick,
1000 sq. ft
9 inches thick,
1000° Fahrenheit difference in temperature.

Taking curve No. 1 we find that at 1000° Fahrenheit difference in temperature that 117 B. T. U.'s are transmitted over each

square foot for each degree Fahrenheit difference in temperature in 24 hours, the wall being one inch thick. The area of the wall is 1000 square feet, its thickness 9 inches, therefore we have

Dif. in temp. Area in sq. ft.

$$= \frac{117 \times 1000 \times 1000}{9 \text{ in.}}$$

13,000,000 B. T. U.'s heat transmitted in 24 hours over 1000 sq. ft. of 9 inch wall.

If the same wall were made of brick whose conductivity is shown by curve No. 4, under the same conditions the heat transmitted would be

Dif. in temp. Area in sq. ft.

$$= \frac{20 \times 1000 \times 1000}{9 \text{ in.}}$$

Thickness in inches

2,225,000 B. T. U.'s heat transmitted in 24 hours over 1000 sq. ft. of 9 inch wall.

or 17.2 percent that of the vitreous brick wall, or in other words 82.8 percent the heat lost through the vitreous wall is retained by the use of brick with lower conductivity.

FACTORS TO BE CONSIDERED IN PORCELAIN INSULATOR DESIGN

BY J. W. WARD.

The rapid development of the modern transmission line with its application of high potentials is demanding of the ceramic engineer a close study of the problems that must be considered in their relation to the class of porcelain that is used for electrical purposes. From a technical standpoint it might be said that the work seems outside of the range of the ceramic engineer, and yet there is a gap that must be closed, and the electrical engineer naturally looks to our society to help close this gap.

There is no item on the transmission line today that calls for more attention than the question of insulation, and such a large part of it is porcelain that the information contained in this paper of some of the factors that have to be considered in the development of a design will no doubt awaken interest in the intricate problems that have to be dealt with. Much progress has been made in the past few years, but there are many important investigations yet to be made.

1. The ideal design for a porcelain insulator is one that will not fail under extreme operating conditions, and if occasion demands, the air surrounding the insulator will break down before puncturing the porcelain. The first point will be governed by the proportions of the mass of material that make up the porcelain, so that internal strains will not be set up in the different manufacturing operations. The second point will be governed by the dimensions or shape of design, so that the porcelain will resist the puncture voltage and at the same time have the required flash-over value.

2. Calculations of design are difficult to make since so many variables enter that affect the results. When the porcelain is used, or tested under different conditions, there are never definitely defined routes of electrostatic flux that one can deal with, how-

ever, each part of the design must carry its share of the duty imposed upon it so that calculations must be verified by judgment of proportions best suited for the design, and experience with results along similar lines.

3. The porcelain design must cover the features that are most desirable for the service in which they are to operate. As a starting point it must be known what the maximum operating voltage will be, the conditions of climate, and where located, geographically. The locality in which the porcelain is used will govern its application, and then should be a clear understanding of these features before a design is recommended for any particular locality, for rain, dust, fog, sea coast, and altitude will all influence the operation.

4. The composition of the body that makes up the porcelain should be dense and homogeneous, and free from injurious manufacturing defects. The glazed surface should be hard and smooth on all surfaces exposed to the weather, and should not be affected by sudden changes in temperature over the range of atmosphere under which it is operated. It should not be affected by nitric acid, nitric oxides, ozone, and it should be moisture proof.

5. Porcelain design can be divided into two classes, single or one piece insulators, and multi-part insulators. The first class covers those that are made up in one part and is usually limited to the lower voltages, except where combinations are made up using the porcelain in multiple or series combinations. The second class covers multi-part insulators where the design calls for shape and size that cannot be manufactured in one piece. They are made up in two or more parts and assembled after the firing operation.

6. The voltage rating or the capacity is very largely based on the voltage necessary to flash-over the porcelain under storm conditions, and the properties that govern these conditions must be considered. In actual service the porcelain is subjected to both mechanical and electrical stresses, the depreciation due to the strains set up by the conductor, and the continual stress set up by the voltage of the line.

7. A porcelain insulator has an actions very similar to a condenser, the effect of capacity plays an important part in the design. The flow of current over the surface of the porcelain is the result of capacity, and in order to increase its efficiency, the electrostatic capacity must be reduced.

The increasing or reducing of the capacity of an insulator will be governed by the shape of the porcelain, the number of petticoats, the flaring of the shells, etc.

8. To provide reliability against puncture or break down in service, the design should provide a safety factor of approximately 2½ to 3 times that of the operating voltage of the line, and this is not always governed by an increase in the thickness of the porcelain parts, but can be controlled by the shape of design, the quality and manipulation of material and the workmanship in manufacturing the porcelain.

9. The American Institute of Electrical Engineers have adopted standard values for sparking distances, in effective volts, between sharp needle points in air. The following figures have been selected from a curve showing the relation between kilovolts in inches.

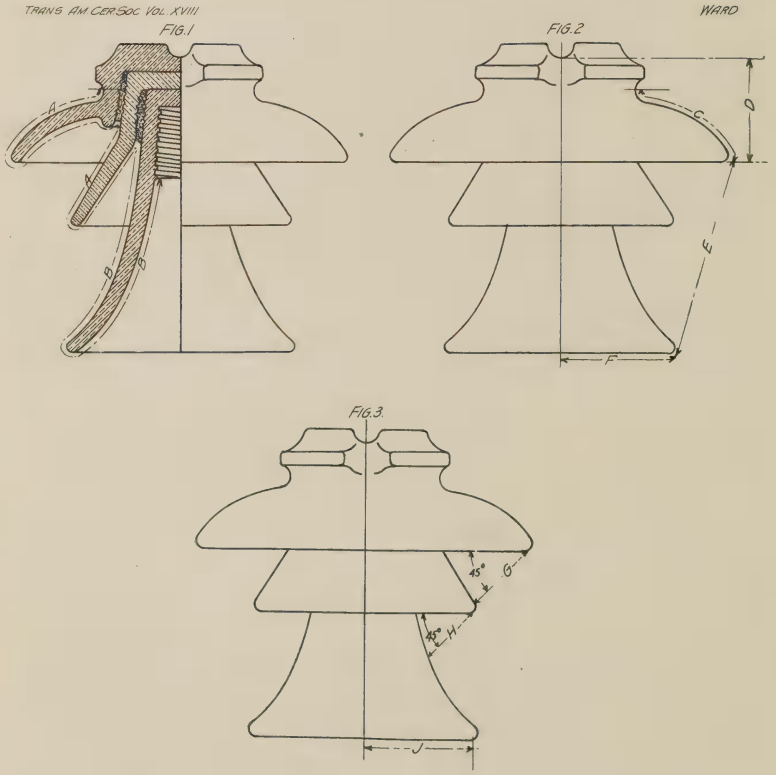
Kilovolts	*Inches*	*Kilovolts*	*Inches*
10	0.47	70	5.85
20	1.00	80	7.10
30	1.625	90	8.35
40	2.45	100	9.60
50	3.55	150	15.00
60	4.65	200	20.50

10. In order to explain how the dry arcing, wet arcing, and leakage distances are established, the outlines shown in Figures 1, 2 and 3 are given and will make these points clear.

11. Leakage distance is the surface of the porcelain measured from the conductor or tie wire to the pin as indicated by the path, in inches, shown as *A-B* in Figure 1.

12. Dry arcing distance is measured, as indicated in Figure 2, by the sum in inches of distances (C or D) $+ E + F$, the distance C or D used depending on which gives the shorter distance between the line wire and the lowest edge of the top shell.

13. Wet arcing distance is measured as indicated in Figure 3 by the sum in inches of $G + H + J$ on a 45 degree line from the edge of one part to the next piece, and from the edge of the center to the center line of a multipart porcelain, and, for actual distance, take ½ of the diameter of the pin to be used, measured on a line at the bottom of the center, and deduct it from the given distance.



14. The wet arcing test is made by producing artificial rain, with the nozzles arranged to have the water strike the insulator at an angle of 45 degrees to the horizontal, for insulators placed in a vertical position, and 45 degrees to the inside of horizontal on strain type insulators. The rate of precipitation should be checked by suitable gauges. The specific rain resistance used

should correspond to the locality, but should not be less than 5000 megohms per cu. in. The rate of precipitation should range from 0.25 to 0.5 inch per minute. The average water supplies are impure, and it is best to use condensed steam, rain water, or melted artificial ice.

15. Arc-over values are governed by the altitude under which tests are made and will decrease with increasing altitudes, or decreasing barometric pressure, and allowance should be made for this condition in calculating or using results from test data. The break-down strength of air varies approximately, though not exactly, as the barometric pressure.

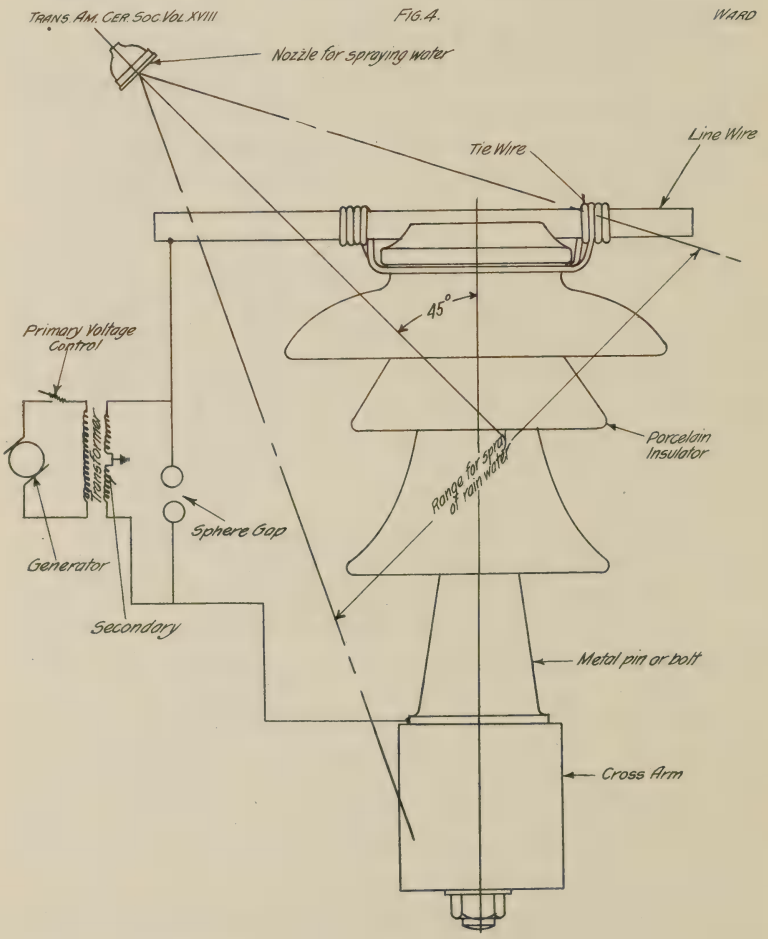
16. Design tests should always cover the dry and wet flash-over values, and should be obtained as nearly as possible in the position that the porcelain will occupy in service. For pin type insulators, they should be mounted on a supporting frame of metal to represent the cross arm and have a metal rod tied in the line wire groove with a piece of bare copper wire to represent the tie wire, and suspended from a metal support for suspension type insulators, having a clamp on the bottom unit to hold the line wire.

The test voltage is gradually applied between the line wire and supporting frame until flash-over occurs. The usual time for design tests is one minute; the porcelain should stand without failure with not less than a dry flash-over of three times the specified line voltage, and a wet flash-over value of twice the line voltage.

17. Puncture test is made by immersing the assembled porcelain parts (or with the meal attachments) in a good grade of transformer oil and applying the voltage equivalent to about 75 percent of the dry flash-over value to start, with a gradual rise of potential until puncture occurs. The value obtained under this test should not be less than 135 percent of the dry flash-over value of the porcelain under test.

18. Figure 4 shows an assembled porcelain, three piece cemented pin type insulator mounted on the cross arm showing the relation of the nozzle for spraying water on the porcelain, also the transformer connections. Figure 5 shows an outline of the porcelain with the location of the rain gauge for checking the rate of precipitation.

19. The mechanical strength features to be considered cover tensile, compression, and torsion tests depending upon the application of the design. On pin type insulators, it is customary to



mount the porcelain on a rigid pin, and apply the pressure at the side tie-wire groove in a perpendicular direction to the vertical axis of the porcelain. As a general rule, pin type insulators will

stand a pull that will bend any metal pin used for this purpose. They should be tested on the pin that will be used in service.

For suspension insulators a tension is applied between the metal cap on the top and the connecting bolt on the bottom. On interlocking type insulators, a compression test is made by applying pressure on the porcelain that separates the cables in practice. The torsion test is applied to designs that will be subjected to this strain in service, and is accomplished by attaching a cap or clamp on the outside shell and a bolt or metal support to the center; the pressure is applied to the outside cap or clamp while the center is gripped rigidly. This test shows up the strength of the material used as a bond in assembling the porcelain parts together.

20. Permanent glaze is a feature in connection with design, for which the ceramic engineer must provide. If the expansion coefficient is very different than the porcelain, it results in crazing of the glaze with temperature variations, and accumulations of dirt, etc., fill the cracks. A smooth, glossy surface prevents easy accumulations of dirt or dust and makes it possible for rain to remove what little may adhere to the surface.

The action of dirt on the surface of the porcelain will reduce the surface resistance and will cause a leakage over this surface. The effective leakage distance over the porcelain surface should be as great as possible without unduly crowding the parts in multi-part insulators.

21. The coefficient of expansion of the metal parts used in connection with porcelain is no doubt responsible for considerable of the trouble experienced in this class of design, though in the majority of cases, it may not be possible to trace it directly to this cause. The data on the expansion coefficient of porcelain, when worked up in the form of a commercial porcelain insulator, brings up always the problem of how it is to be obtained.

The figures generally used for the coefficient of expansion of porcelain are from .000,004,5 to .000,006,5 between 20 degrees and 250 degrees Centigrade while the figure used for M. I. iron is, 0.000,008,99.

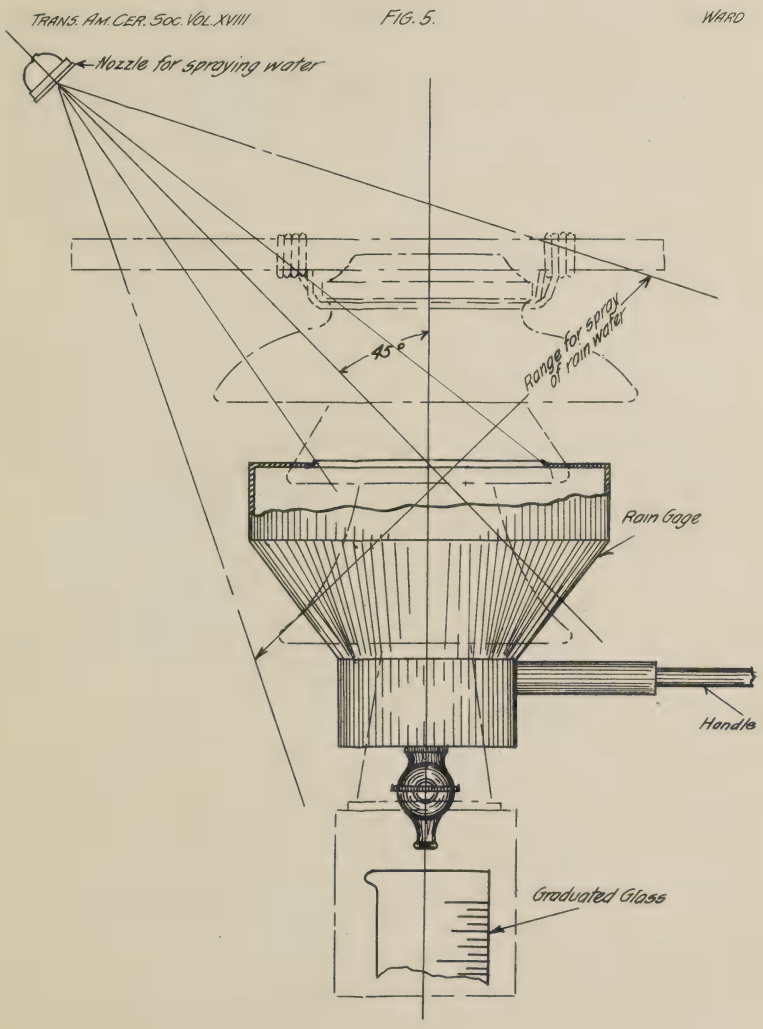
22. The sudden variations in temperature to which the porcelain is subjected in service has suggested a hot and cold test,

especially on designs calling for metal and porcelain combinations. This is accomplished by immersing the complete porcelain in boiling water, then in ice water. After a number of transfers from hot to cold water, the porcelain is tested electrically, and this will usually show up any faults that are developed by the test. To locate strains or cracks that may be developed, a test can be made by putting the porcelain in a solution that will penetrate the flow and stain the surface, where (when the electrical tests are made) one can follow the path of failure. A limit for the hot and cold test can be established by using the extreme ranges in temperature under which the porcelain will be used.

23. Dew tests are usually confined to porcelain insulators that are used in tropical countries and is called for on account of the rapid temperature changes which cause a heavy dew to form on the porcelain. This test is arrived at in a variety of ways, the method usually employed is to allow free steam to blow on the porcelain until it is covered with moisture.

24. Simultaneous mechanical and electrical tests consist of subjecting the porcelain to a mechanical strain, while at the same time under electrical pressure. The test voltage is held constant at just under the flash-over voltage, while the mechanical pull on the porcelain is started at a point considerably below its breaking point and gradually raised in steps of 1000 or 2000 pounds. The porcelain should not fail electrically at less than approximately 2½ times the maximum pull that it would be guaranteed for in operating conditions.

25. Compressive strength tests on representative porcelain insulator bodies, glazed and unglazed, show that unglazed porcelain will stand a higher breaking load than glazed porcelain. There are many variables to be watched, if accurate results are to be obtained. It is necessary to provide even bearing surfaces, and if grinding is resorted to, it must be done carefully or the surface will have a formation very similar to a glaze, and this will cut down the average breaking point. It is a good plan to use a thin sheet of some free flowing material such as sheet lead approx. imately 1/32 inch thick, which will add slightly to the strength, but will furnish a more even bearing surface.


TRANS. AM. CER. SOC. VOL. XVIII
FIG. 5.
WARD
Nozzle for spraying water
45°
Range for spray of rain water
Rain Gage
Handle
Graduated Glass

26. Porcelain and metal parts assembled together have, as a rule, a bond of cement, and experience so far shows that a good grade of neat Portland 80 percent cement mixed with 20 percent pure water, by weight, gives a good bond material. Careful tests on the cement should be made for fineness of grain, specific gravity, tensile strength and constancy of volume. Care must be exercised in the mixing of cement, and it should be handled carefully to avoid any air bubbles when assembling the parts. Only mix as much cement as can be used up in not over 25 minutes. It is a good practice to keep the assembled parts in moist air for at least 24 hours after cementing, and they should not be subjected to mechanical tests under 7 or 8 days.

27. The mechanical loads applied to porcelain in service affect the life of the insulator, and have considerable to do with its reliability. The mechanical requirements must be considered in the porcelain design, as high working loads affect the electrical factors of safety to such an extent that the two combined will cut down the factors of safety and reduce the dielectric strength of the porcelain.

28. In assembling of porcelain parts with metal parts some method should be employed to counter-balance the stresses set up by different temperature expansions of the different materials. The following are some materials that have been used — a thin layer of felt, or paraffine-cork approximately 1/16 in. thick, also the use of a thin layer of elastic plastic varnish. These materials have enough elasticity to allow for some of the stresses set up by the porcelain and metal, and by the swelling of cement.

29. A critical feature to be watched in design is sharp angles, and the number and shape of grooves for cementing of the parts together. These points will have a tendency to set up strains in manufacturing that are not fully developed and do not always show up readily, but when the extreme sudden operating stress is applied, due to say, a lightning discharge, there is eventually a breakdown of the porcelain that can be traced directly to these points. Recent practice has been to allow plenty of radius on all angles so as not to set up strains at these points, and to eliminate grooves as much as possible, resorting to a sanded surface for gripping on the bonded material between different parts.

The sanding of the surface has the advantage of holding the parts together equally well for tests in torsion or tension and eliminates the grooves giving additional thickness and dielectric strength to the porcelain.

30. The absorption test of porcelain insulators will show up porous material. The class of porcelain which shows high absorption, will depreciate very rapidly in service, and soon lose its power to resist the electrical pressure applied, due to the absorption of moisture which lowers the resistance of the porcelain.

There are several methods used in making this test as called for by different purchasers; usually an ink test is specified, but this is not very satisfactory. Unless the material is so porous that observation would leave no doubt on this point, many conditions make it almost impossible to use the ink test as a basis for comparison. It is a hard matter to wash off the surface the colored ink used, and when the test piece is broken as a rule, some signs of the coloring fluid will be found in the fracture, not through absorption, but by following the surface in breaking the porcelain.

A more reliable test is immersion in water for a specified length of time. The absorption is calculated on a percentage basis of increase in weight. When broken and submerged in water for 100 hours the porcelain should not absorb more than ½ of one percent of its weight.

31. The law of corona must be considered in porcelain design as that point at which the air begins to fail as an insulator, and the escape of energy through the air begins as corona. The porcelain should not show a corona loss at normal operating pressure. The action of corona is manifested either chemically, mechanically, or by heat. If the corona discharge is only moderate, it is accomplished by the production of ozone from the surrounding air, and this may cause a sort of a chemical action. If usually severe, it may cause a chemical combination of the oxygen, nitrogen, and water vapor of the air. The mechanical action of corona seems to be of a bombarding nature, having a tendency, when the porcelain is subjected for long periods of time, to drill minute holes in the surface of the porcelain.

NOTE ON THE DETERMINATION OF SEVERAL DISSOCIATION POINTS

E. T. MONTGOMERY AND M. M. GROVES, ALFRED, N. Y.

The results here recorded are technologic rather than purely scientific and have for their purpose the bringing out of practical applications more clearly, if possible. Though the considerations of pure science, thus far developed, were kept in mind, it was the aim of the writers to avoid the complicated methods necessary, when all scientific precautions are observed, and reduce these to as near practical conditions as possible, except that very small samples were used so that equilibrium could be reached in the shortest possible time. To illustrate this point — the true dissociation point of calcium carbonate is that temperature at which the vapor pressure of the CO_2 present, reaches one atmosphere; or in other words, that temperature at which the carbonate can be completely dissociated into CaO and CO_2 in an atmosphere of pure CO_2 under 760 mm. pressure. Calcium carbonate in most ceramic operations does not dissociate under these conditions, hence the required temperature will vary considerably from the theoretical.

For all of the work discussed in this paper, the loss in weight method was used, the weighing being done directly, without removing the sample from the furnace. The apparatus used is shown in Figure 1 and is almost self explanatory. The furnace is of the platinum wire resistance type with a solid bottom. The sample is supported in the furnace in a small platinum foil capsule, one-half inch in diameter, to which a short length of platinum wire is attached. One pan of the delicate chemical balance mounted above the furnace is replaced by a bent and counterbalanced steel wire of equal weight. This bend in the wire at the point where it passes through the box in which the furnace is located, is for the purpose of avoiding any direct openings from the furnace to the balance, through which rising air currents

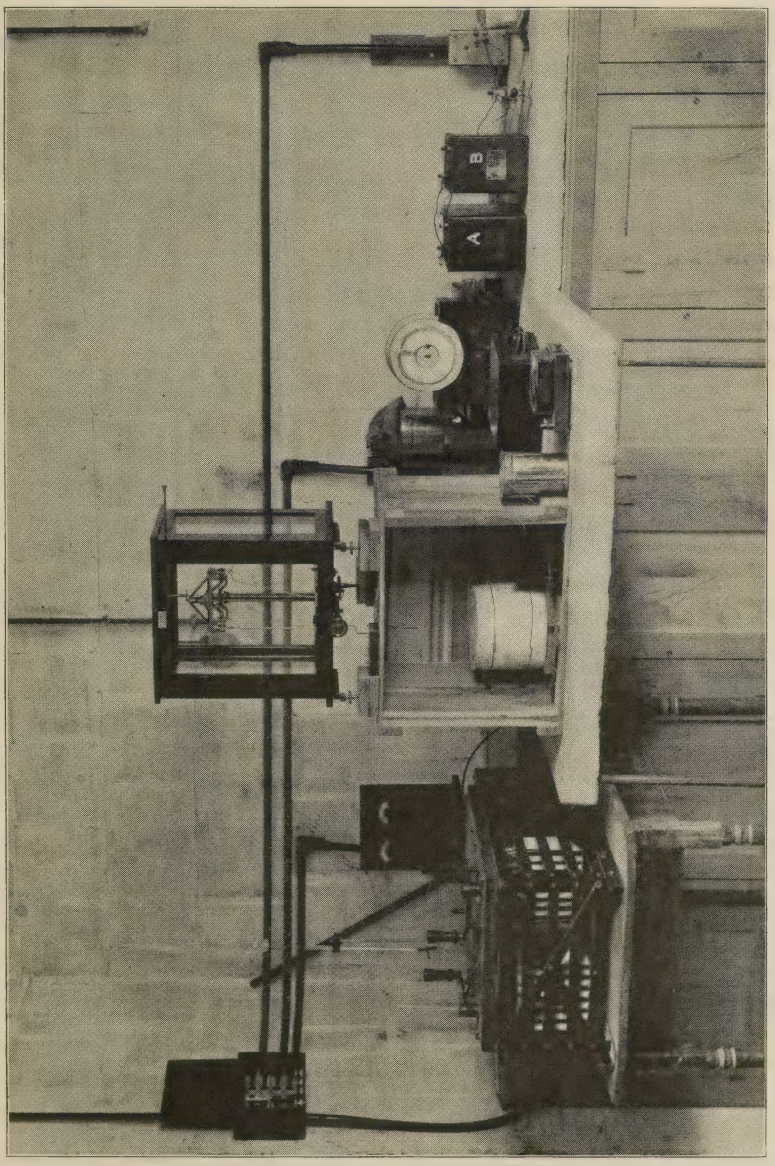


FIG. 1.

could be carried. The steel wire terminates in a hook from which the platinum capsule is suspended.

The mouth of the furnace was closed with a fired clay plug or cover in which there were two openings of considerable size, one for the wire supporting the capsule and one for the thermocouple. The junction of the couple was placed near the bottom and just outside of the capsule. In none of the experiments was the atmosphere inside the furnace changed or conditioned in any way. The top was sufficiently open so that no pressures could develop, and enough circulation could take place to keep the atmosphere in the furnace practically the same as that in the room. We are now ready to consider the several determinations.

Dissociation of Calcium Carbonate. The first determination is that of the dissociation temperature of $CaCO_3$. Johnson,[1] by the vapor pressure method, plotting a curve, and extrapolating on that curve, determined that in the dissociation of $CaCO_3$, the CO_2 pressure reaches one atmosphere at 898° C. By lag curve methods, Bleininger and Emley[2] at the Pittsburgh laboratory of the Bureau of Standards have determined that in 37 samples of limestones the dissociation point was clearly indicated at 880° C. This temperature has also been checked at Alfred on the same type of sample and using the same method.

Using a powdered sample of Baker's analyzed, precipitated $CaCO_3$, weighing only .1830 gram, in the apparatus just described, the writers were able to get a clearly indicated loss in weight at 620° to 627° C. and holding this temperature, the loss in weight was found to be at the rate of 2.35 percent per hour. As it would have taken a longer time than could very well be arranged for in order to get off the full 44 percent of CO_2 present even if this rate held until the end of the experiment, the temperature was raised to 647° C. At this temperature the rate of loss in weight became about 4 percent per hour. The sample was now held at this temperature for 15 hours until a total of 44.00 percent CO_2 had been driven out, and the sample had become constant in weight as a check on the determination. The rate of loss gradually decreased to less than 1 percent per hour. Of course, the vapor pressure

[1] J. Johnson, *Jour. Am. Chem. Soc.*, Vol XXXII-8-1910.
[2] *Trans. Amer. Cer. Soc.*, Vol. XIII, p. 618.

of the CO_2 has a definite value at all temperatures, but from the nature of the reaction it would seem to be entirely feasible to drive off all of the CO_2 at a temperature as low as 620° to 630° C. under the conditions of the experiment within a reasonable length of time. In other words, we can look for the active dissociation of $CaCO_3$ to begin at about 620° C. in such ceramic operations as the fritting of glazes or the burning of cement. The temperature will be considerably higher in lime burning because of the size of the lumps of carbonate and the high content of CO_2 gas in the kiln atmosphere. This experiment is, of course, not scientifically exact because an atmosphere of pure CO_2 under 760 mm. pressure was not maintained in contact with the carbonate. The mean barometric pressure at Alfred is 720 to 740 mm. Some CO_2 was, of course, present in the furnace during the entire experiment, as there were no currents of air passing through the furnace which would carry out the heavy, dissociated CO_2 gas. There must, therefore, have been developed a pressure of 720 to 740 mm. at 620° to 647° C. in an atmosphere containing a small percentage of CO_2, while Johnson reports a temperature of 898° C., or very near that, as necessary to reach this pressure in an atmosphere of pure CO_2.

A cold junction was not used on the thermocouple in this experiment, but all of the temperatures given are the corrected temperatures.

There are many objections to be found with vapor pressure methods such as those used by Johnson, Pott and Zavriev. Some of these Johnson pointed out, but even in his more exact apparatus many faults still remain. One must use small quantities in order to get a uniform temperature throughout the sample and to avoid errors of temperature measurement. Under such conditions small amounts of vapor or other impurities capable of developing a pressure lead to erroneous results. Also the friction of the mercury in the trap and manometer tubes is very great in comparison with the small pressure developed from a small sample. It is also difficult to know when equilibrium conditions have been fully established, as erratic pressures are almost always encountered at unexpected places. It is often a question in working with complicated apparatus whether we do not frequently intro-

duce greater errors than those we seek to avoid. The senior writer contemplates further work on the determination of the true dissociation point of $CaCO_3$, using this simple loss in weight method by the expedient of enclosing the whole apparatus in a gas tight case and maintaining in this case an atmosphere of CO_2 at 760 mm. pressure.

Dissociation of Calcium Hydrate. The second determination to be discussed is that of the dissociation or dehydration temperature of calcium hydrate, $Ca(OH)_2$, commercially known as hydrated lime. There are no ceramic operations which the writers can think of at present in which this reaction takes place, but it is of interest nevertheless, and the data may some day be of practical as well as of theoretical value. It was a practical consideration, however, which lead to the study of this compound. The phenomenon of "burning" or "scorching" in the slaking of quick lime, where a deficiency of water has been used, has not yet been explained. This scorched lime is known to form hard lumps, to slake very slowly, and is considered by most contractors and other users as ruined and valueless. It was thought by the writers that the cause of this trouble might be due to the temperature reaching the dissociation point of the hydrate, under the conditions which cause scorching, thus possibly producing an inert form of the hydrate which was chemically and perhaps physically different from the rest of the mass.

This theory proved to be unfounded in fact. At least no evidence was found to support it in the limited study made of this point. It was found that $Ca(OH)_2$, dehydrated at 500° C., will rehydrate and slake with almost violent rapidity.

Coming back to the determination of the temperature at which this dissociation takes place, R. K. Hursh[3], after discussing quite fully, the work done by other investigators previous to his experiments, states as the result of his work on this compound, that two dissociation points were found, one one at 375° C. and a second at 580° C. Johnson[4] has found the dissociation pressure of $Ca(OH)_2$ to reach 760 mm. at 547° C. It should be borne in mind that this dissociation point must, theoretically, be determined

[3] *Trans. Amer. Cer. Soc., Vol.* XIV, p. 792.
[4] *Zeitschrift fur Physikal Chemie,* Vol. 62, p. 330.

in an atmosphere of pure air, saturated with moisture at the temperature of the experiment, and under a pressure of 760 mm.

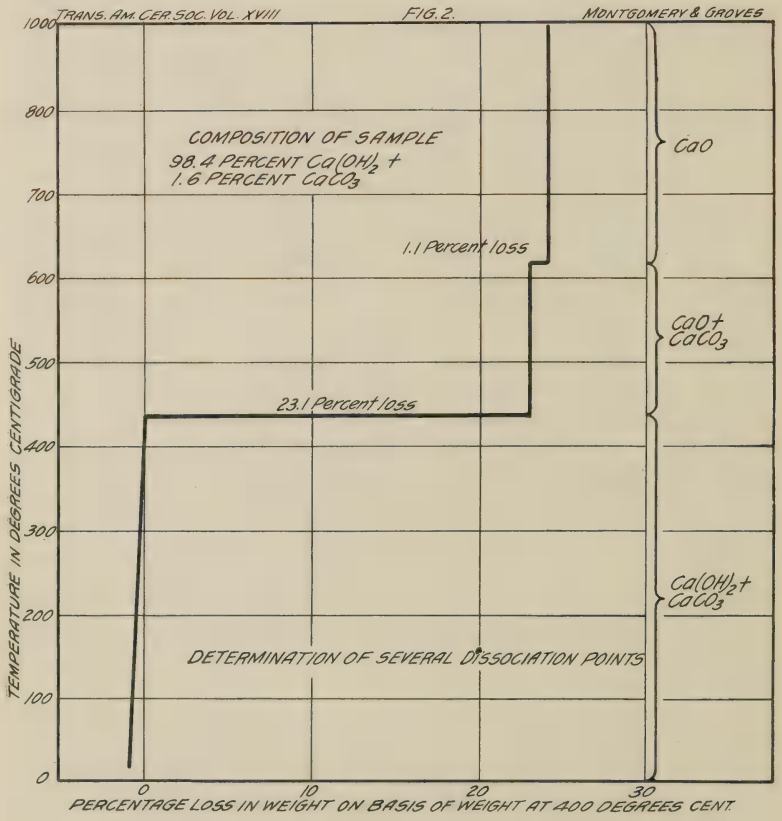
Making this determination in the apparatus and under the conditions previously described, the following results were obtained. Only one dissociation point was found for the hydrate, namely at 430° C. to 439° C. In all of some twelve check determinations a clearly defined loss in weight occurred at this temperature, and, by holding the temperature, complete dehydration could be accomplished in a reasonable length of time. The dissociation of the $CaCO_3$ present was found to always occur at 617° C. on raising the temperature after completing the dehydration of the hydrate and obtaining constant weight.

For these tests, samples were prepared in two different ways in order to obtain, if possible, a sample in which the crystalline hydrate would predominate and also a sample in which the hydrate would be largely amorphous. Sosman[5] has shown that pure lime, CaO, is obtainable in two forms. The first form which results from the dissociation of $CaCO_3$ at low temperatures is very fine grained, porous and probably amorphous. On heating this porous lime to higher temperatures, or dissociating $CaCO_3$ at high temperatures, it gradually changes over to the second form, a cubic crystalline lime of refractive index 1.83. Klein[6] has shown that there is both a crystalline and an amorphous form of the hydrate. The fine, low burned, amorphous lime tends to produce, on hydrating, the amorphous form of the hydrate, while the coarser, high-burned lime, produces a preponderance of the crystallized hydrate.

One sample was prepared by burning a pure white marble to 1300° C. and the second by dissociating Baker's C. P. precipitated $CaCO_3$ at 700° to 740° C. The CaO thus obtained was slaked in an excess of distilled water, and the excess H_2O absorbed in a desiccator. Each sample was finely pulverized for use and analyzed for CO_2. From this the percent of $CaCO_3$ present in the samples was calculated. Through the kindness of the Bureau of Standards Laboratory at Pittsburg these samples were examined by Mr. A. A. Klein, whose report shows that the object

[5] *Jour. of Wash. Academy of Sciences*, Vol. 5, No. 16, 1915.
[6] *U. S. Bureau of Stanards*, Technical Paper No. 43, pp. 58-60.

in view was not attained, but that both samples consisted chiefly of amorphous hydrate. This report was as follows: "The hydrated lime sample from the low burned lime consisted of amorphous hydroxide with a small amount of crystallized hydroxide and calcium carbonate. The sample of hydrate from the high



burned lime consisted mainly of amorphous hydroxide with a small amount of crystallized hydroxide, calcium carbonate and a trace of free lime."

Various weights of sample were tried in different determinations, but a sample weighing about one-half gram was found to give the most satisfactory results. In the case of the hydrate

from the high-burned lime, a slight and gradual loss in weight amounting to about 1 percent was observed up to a temperature of 400° C., at which temperature the weight became constant. This was taken to indicate the presence of a small quantity of mechanically held water, since either rapid or slow heating up to this temperature made no appreciable difference in the loss. There was no indication that the hydrate was being dissociated, so the weight at 400° C. was taken as the basis for calculating the loss of combined water from the hydrate, and CO_2 from the small amount of carbonate present. In the case of the hydrate from the low-burned lime, a slight increase in weight occurred between 300° and 400° C. due to an absorption of CO_2 or moisture from the air. This gain was subtracted from the weight at 400° C.

Upon raising the temperature slowly and allowing plenty of time to reach equilibrium, the lowest temperature at which dissociation of the hydrate was indicated was at 430° C. for the hydrate from the low burned lime and at 439° C. for that from the high-burned lime. $Ca(OH)_2$ contains 24.32 percent of water. By holding the temperature at the dissociation point, it was possible to drive out an amount of H_2O equal to 23.5 percent of the $Ca(OH)_2$ present in the sample. This is about as close to the theoretical as is practically possible. After the sample became constant in weight at this temperature, the temperature was gradually raised until a second loss occurred at 617° C. Holding this temperature, a loss occurred approximately equal to the CO_2 present in the sample. No further loss occurred on carrying the temperature on up to 1000° C. These results are shown graphically in the accompanying curve, Figure 2. All of the temperatures given are cold-junction thermocouple readings. The barometric pressure was between 720 and 740 mm.

Dissociation of Gypsum. A third dissociation temperature studied was that for gypsum, $CaSO_4 . 2H_2O$, the raw material of the plaster industry. We have long been told that by heating this dihydrate ($CaSO_4 . 2H_2O$) for a considerable length of time at 107° C. or a little above, we have formed, the hemihydrate ($CaSO_4 . \frac{1}{2}H_2O$), commercially known as plaster of Paris, stucco plaster, pottery plaster, or dental plaster, depending on the purity and fineness.

Based on the results of recent experiments, the writers venture the statement that there is no such definite compound as the so-called hemihydrate, $CaSO_4 . \frac{1}{2}H_2O$.

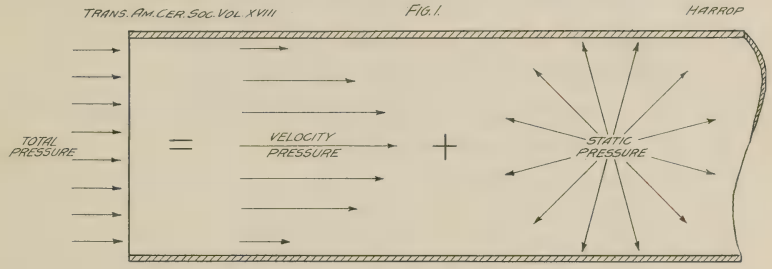
Gypsum, $CaSO_4 . 2H_2O$, contains 20.9 percent of water. The sample used in the determination of its dissociation or dehydration point was prepared by pulverizing pure gypsum crystals to pass a 100 mesh sieve and drying the powder in a desiccator. A small portion was placed in the capsule and weighed at room temperature in the furnace. The weight was .2920 gram. It lost nothing in raising the heat to 100° C. At 105° C. a loss in weight was noted and holding the temperature constant between 105° and 107° C. for 4 hours an amount of water was driven off equivalent to 20.7 percent of the weight of the sample, thus giving a completely dehydrated product at this temperature. After the reaction was completed, the weight became constant and no further loss occurred on raising the temperature. These temperatures were measured by means of a thermometer checked by calibration in steam over boiling water, the boiling point being calculated for the barometric pressure read that day.

THE INADEQUACY OF STATIC PRESSURE DRAFT GAGES

BY C. B. HARROP.

A "good draft," as indicated on an ordinary draft gage, usually satisfies the clay worker that his chimney is doing satisfactorily the work for which it was intended.

The real work that a chimney is doing, the principles which underlie its operation and especially the actual meaning of draft gage readings, are understood by comparatively few men. Even conceptions of the meaning of the word draft, as applied to chimneys, vary considerably, but not in any degree comparable



with the variations which are common in the draft readings themselves.

It is assumed here that the reader understands why hot gases rise in a chimney.

Physics of Gas Flow. Considering the physics of the flow of gases through pipes or flues, we find that in order to have a flow, we must have a pressure, or head. This total pressure, which is responsible for the flow, serves two purposes: First, to create the velocity of flow, and second, to maintain this velocity of flow against the resistance offered. The pressure serving the first purpose is termed the velocity pressure and that serving the second is known as the static pressure, the latter, however, is **not**

necessarily the pressure measured by the ordinary or static-pressure draft gage.

It is thus seen that at the bottom of a chimney, which is operating, we have two separate and distinct pressures, — one represented in the moving gases and acting in the direction of their flow, greatest in the center and least at the perimeter, and another pressure acting in all directions and theoretically equal throughout the cross section of the chimney at any particular level.

Total Draft Pressure or Draft Intensity. Another and more common expression for the total pressure, as used in connection with chimneys is draft intensity, which will be found greatest at the base, will be reduced to half, midway to the top, and at the outlet will fall to zero or will be equal to atmospheric pressure.

The draft intensity may be figured from the following formula:

(1) $F = 0.192 \times H \ (D - d)$

where F = intensity of draft or unbalanced pressure in inches of water column.

D = density or weight of outside air in pounds per cubic foot.

d = density of weight of chimney gases in pounds per cubic foot.

H = height of chimney in feet.

0.192 = height of water column in inches equal to one pound pressure per square foot.

The density of a gas varies inversely with its absolute temperature,

(2) therefore $D = \frac{r}{r_1} \times 0.0807$

(3) and $d = \frac{r}{r_2} \times 0.085$

where r = absolute temperature at 32° F. = 491°.
r_1 = absolute temperature of external air.
r_2 = absolute temperature of chimney gases.
0.0807 = weight of one cubic foot of air and 0.085 = weight of average flue gases, both expressed in pounds at 32° F.

Substituting (2) and (3) in (1), we have

$$(4) \quad F = 0.192 \times H\left(\frac{491}{r_1} \times 0.0807 - \frac{491}{r_2} \times 0.085\right)$$

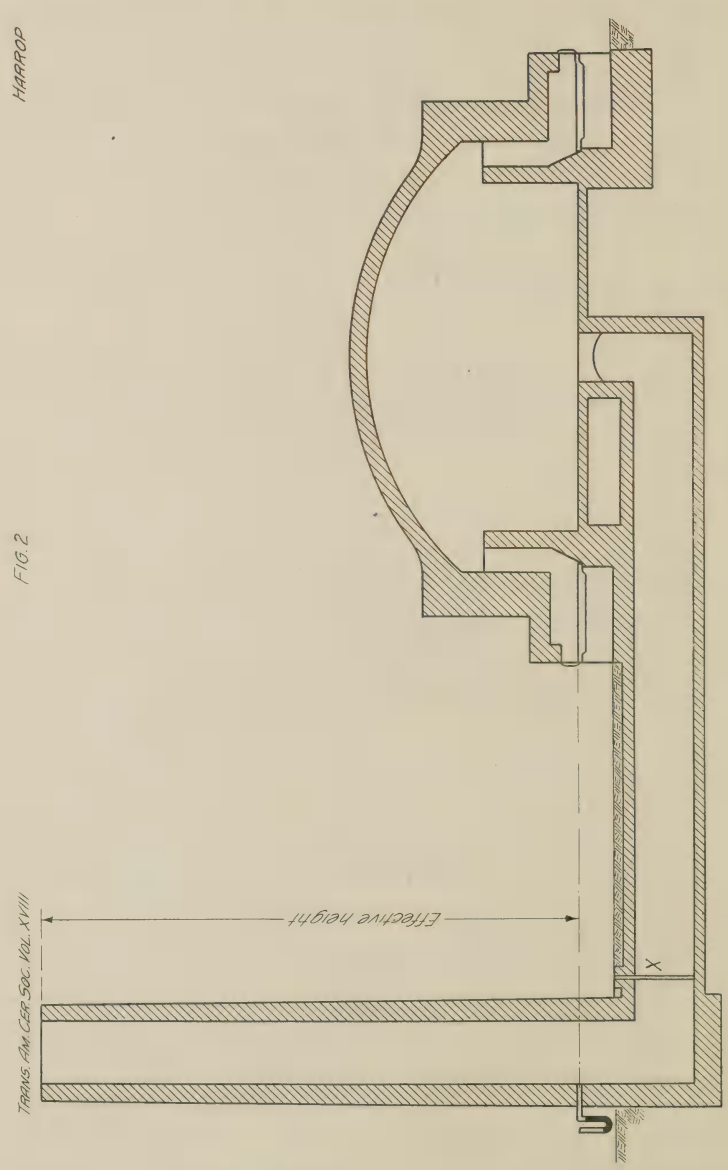
If we assume a chimney 50 feet high and the temperature of the outside air at 70° F., the above formula becomes

$$(5) \quad F = 0.718 - \frac{401}{r_2}$$

Curve A, Figure 3, shows the variations in draft intensity at various intervals above an atmosphere temperature of 70° F.

Inconsistencies in Static Draft Gage Readings. Let us assume a chimney with a grate bar furnace built into its base and with a temperature of internal gases of 600° F. If fire and ash doors are closed, the draft intensity will be say 0.6 inches water column. If the ash doors are opened and we consider the friction in the chimney as negligible, a velocity pressure of say 0.3 inches is developed and we would have a draft reading of 0.6 — 0.3 = 0.3 inches over the grates. Assuming that the chimney temperature remains constant and that the fuel bed is made thicker, so that it offers 50 percent more resistance to the passage of air, then only two-thirds as much air will pass through, and the corresponding velocity pressure will be two-thirds of 0.3 or 0.2 inches. Then the draft reading would be 0.6 — 0.2 = 0.4 inches.

Thus with the ordinary draft gage we have 0.3 inches draft and upon thickening the fuel bed and thereby allowing less air to pass through the grates with a corresponding decrease in the rate of combustion, the gage reads 0.4 inches, indicating a better draft which is entirely erroneous. Of course, the chimney temperature may soon fall, as will also the draft gage reading, but the amount


HARROD
FIG. 2
TRANS. AM. CER. SOC. VOL. XVIII
Effective height
X

of drop in the gage will not correspond to the reduction in velocity or volume of gases passing up the chimney.

On the other hand, suppose that the fuel bed becomes thinner, the resistance less and the velocity pressure of 0.3 inches thereby is increased 33 1/3 percent. Then the draft reading would be $0.6 - 0.4 = 0.2$ inches. Thus our gage shows a reduced draft, while the actual velocity (and therefore volume) of gases passing up the chimney has been increased 33 1/3 percent.

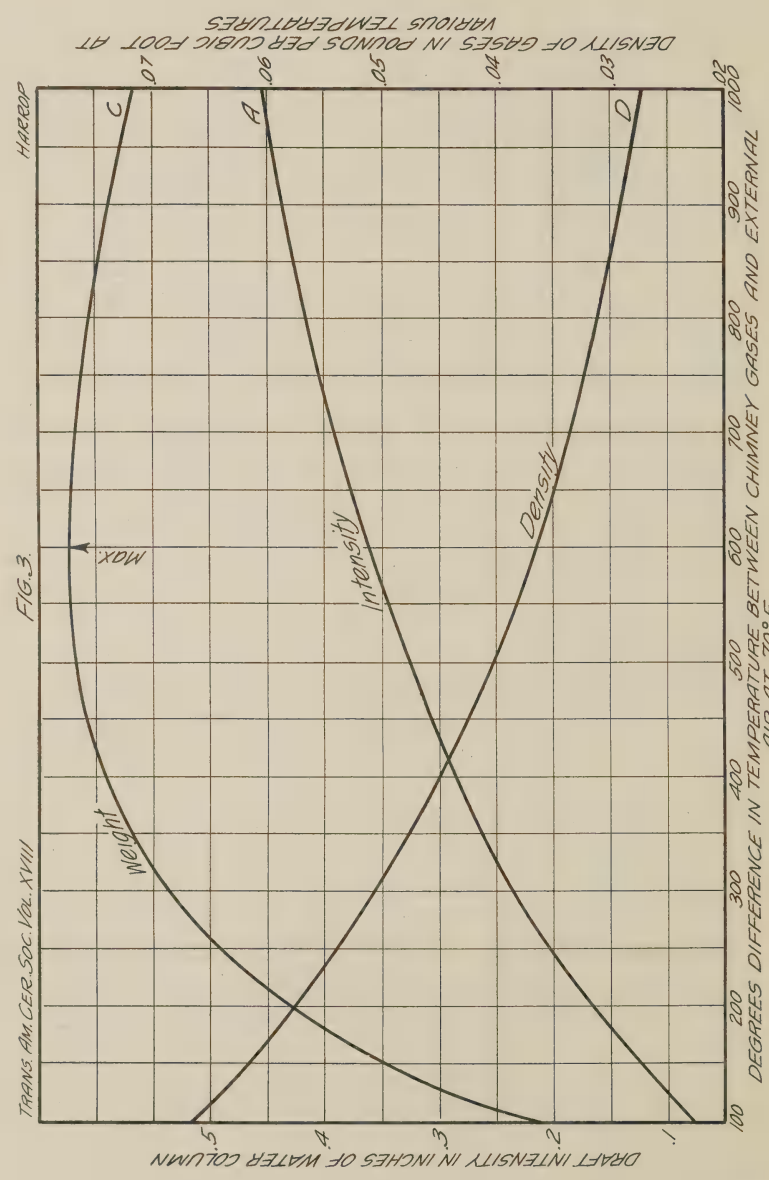
To illustrate further the false ideas regarding draft in kiln chimneys, let us assume a kiln with a single flue leading to an outside chimney (Fig. 2). There is a vertical damper in the flue close to the chimney, and a draft gage is inserted at the grate bar level.

If the resistance is made infinite by closing the top of the chimney or by closing the damper at X, the velocity pressure becomes zero and the static draft reading becomes equal to the draft intensity or total draft pressure, which will be a maximum in the lowest part of the flue system. However, when the furnaces are open and the kiln and chimney are operating, only that part of the chimney above the grates is effective in producing a velocity.

In the early part of the burn, the furnaces and damper are wide open, in an endeavor to secure a large circulation of air through the kiln for the removal of water vapor during water-smoking. During the oxidation period the chimney becomes well heated, and the total draft pressure, while only moderately high, is used largely in producing velocity, leaving a moderate draft to be indicated on our gage.

During the vitrification period, when the chimney is very hot and its power is maximum, we damper it and close the furnaces for heat-soaking. This reduces the velocity pressure and therefore volume of gases going up the chimney, but these gases are very hot and maintain the chimney temperature. Thus with a decreased chimney velocity, the static gage shows a higher draft.

In addition to the inconsistencies already described, a very great source of error in measuring what is supposed to be draft lies in the manner in which the draft tube is inserted into the chimney or flue. As the static pressure is assumed to be the same throughout the cross section at any level in the chimney, the draft


DENSITY OF GASES IN POUNDS PER CUBIC FOOT AT VARIOUS TEMPERATURES
.07
.06
.05
.04
.03
.02
Hazard
C
A
D
Intensity
Density
Weight
MAX
FIG. 3.
TRANS. AM. CER. SOC. VOL. XVIII
DRAFT INTENSITY IN INCHES OF WATER COLUMN
.5
.4
.3
.2
.1
100 200 300 400 500 600 700 800 900 1000
DEGREES DIFFERENCE IN TEMPERATURE BETWEEN CHIMNEY GASES AND EXTERNAL AIR AT 70° F

tube may be inserted through the wall into a small pocket in the inner brick surface, so as not to be in the direct path of the gases. If the end were cut square, the tube could extend into the shaft, providing the opening is very small and the tube is placed at right angles with the direction of flow of the gases. If in the case of a chimney, it is allowed to point slightly downward, the velocity pressure will reduce the draft reading to some degree, depending upon the angle. If the tube points upward, the rushing of the gases past the opening will produce a suction, which will result in a larger and false static reading.

Maximum Weight of Gases Discharged Indicates Maximum Draft. As will be noted from curve D, Figure 3, the density of chimney gases decreases as the temperature increases. Also it will be noted that the draft intensity increases indefinitely with the temperature. The products of the intensity and the density at corresponding temperatures give a curve C, representing the weights of gases discharged. On the assumption that there is no resistance offered to the flow of gases, the maximum weight of gases discharged occurs at a temperature of about 600° F. above atmospheric temperature.

Not only is the static-pressure draft gage — whether it be U-tube, inclined or other differential type — very apt to give incorrect static pressure readings, but the measurements which it indicates, are not what we actually want. This negative static pressure of chimneys, taken at one place only, is of no particular value. The thing with which we are concerned in connection with draft is the velocity of the gases, and as has been shown above, the static draft gage, even if properly applied, takes no account of this. On the other hand, it will under certain conditions show a low draft when the velocity is high and a high draft when the velocity is low.

If it is desired to determine the velocity of gases at various internal temperatures in a chimney 50 feet high and 3 feet square inside, atmospheric temperature being 70° F. and the only resistance offered to the movement of the gases being the friction against the inside walls, we can make use of the following formulæ,

where V = velocity of chimney gases in feet per minute,
H = height of chimney in feet,
g = acceleration due to force of gravity,
h_v = head in feet of chimney gases required to produce the velocity V,
h_f = head in feet of chimney gases required to maintain the velocity V,
h = head in inches of water required to maintain the velocity V,
h_t = head in feet of chimney gases required to produce and maintain the velocity of V,
s = total square feet of surface in chimney offering resistance to the flow,
k = resistance offered by one square foot of chimney surface to gases flowing at the rate of one foot per minute,
a = area of chimney in square feet,
t_c = chimney temperature in degrees F.,
t_o = atmospheric temperature in degrees F.,
d_w = weight of one cubic foot of water,
d_g = weight of one cubic foot of chimney gases.

$$(6) \quad V = 60 \sqrt{2\ g\ h_v}$$

$$(7) \quad h_t = h_v + h_f \text{ or } h_v = h_t - h_f$$

substituting (7) in (6),

$$(8) \quad V = 60 \sqrt{2\ g\ (h_t - h_f)}$$

$$(9) \quad h_t = \frac{H\ (t_c - t_o)}{459 + t_o}$$

$$(10) \quad h = \frac{K\ S\ V^2}{5.2\ a}$$

$$(11) \quad h_f = \frac{h\ d_w}{12\ d_g}$$

substituting value of h from (10) in (11),

$$(12)\quad h_f = \frac{d_w\ K\ S\ V^2}{12\ d_g\ 5.2\ a}$$

substituting values of h_t and h_f from (9) and (12) in (8),

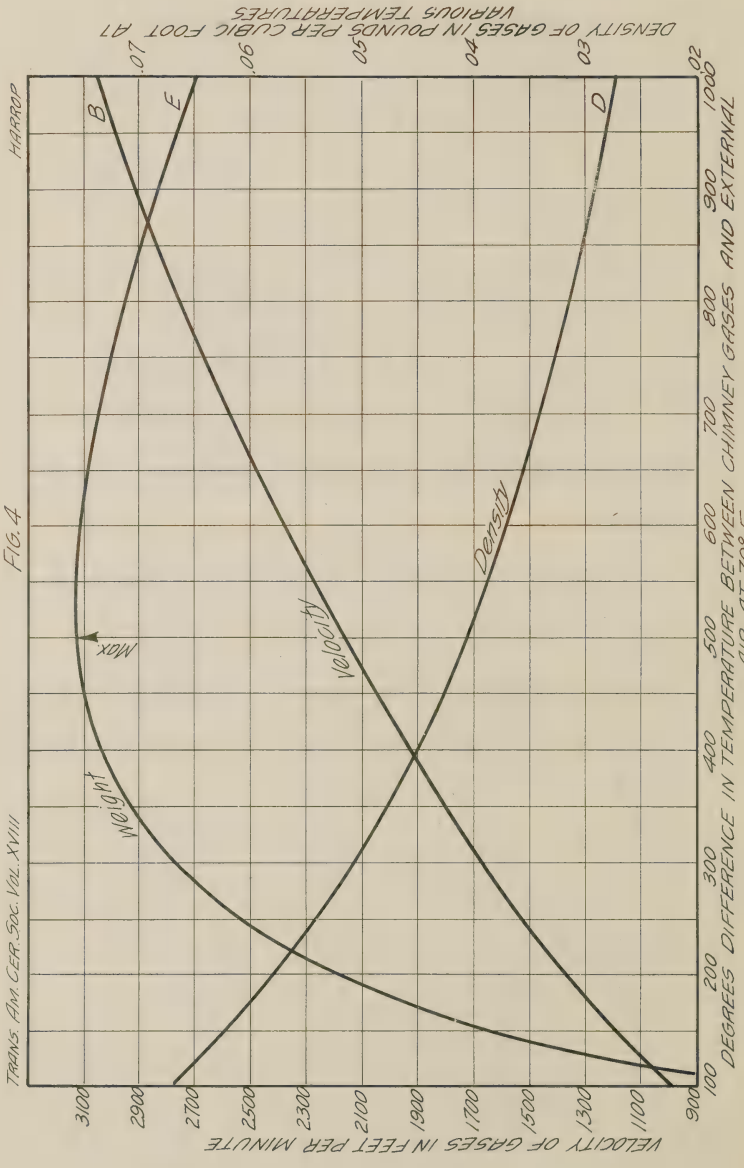
$$(13)\quad V = 60\sqrt{2\ g\left\{\frac{H\ (t_c - t_o)}{459 + t_o} - \frac{d_w\ K\ S\ V^2}{12\ d_g\ 5.2\ a}\right\}}$$

From this formula we can calculate the velocity of chimney gases at various temperatures, as indicated by curve B, Fig. 4. The products of the velocity and density at corresponding temperatures give a curve E, representing the weights of gases discharged against the friction of the chimney. Maximum draft by this method of calculation occurs at about 500° F. above atmospheric temperature.

The scale to which the weight curves C and E are plotted is not indicated.

A Graphic Study of Draft Pressures. Fig 5 Let W represent a tank of water with a pipe of uniform diameter connected horizontally near the bottom, having a valve **X** near the end. Vertical pipes **A, B** and **C,** open at the top, are connected with the horizontal pipe at regular intervals. If the valve is closed, the top of the water will stand level in tank and vertical pipes — height **h** representing the head which produces the maximum pressure, which will be found in the horizontal pipe.

If the valve is opened to the full size of the horizontal pipe and the height of water in the tank maintained, the heights of water in the vertical pipes will assume the positions indicated by the inclined line. Under these conditions, we find at **y** the head or total pressure responsible for the flow and which is the sum of both velocity pressure **V** and static pressure S, — the static pressure being a measure of the fractional resistance from this point to the outlet. At **a,** we find the same velocity pressure but a lower static pressure, because the resistance between this point and the outlet is less, *i. e.* the static pressure as indicated by the height of the water in the several columns becomes less towards the outlet, where we find it to be zero. As the velocity pressure is uniform throughout the length of the horizontal pipe and the


HARROP
FIG. 4
TRANS. AM. CER. SOC. VOL. XVIII
B
E
D
Weight
Velocity
Density
Max
VELOCITY OF GASES IN FEET PER MINUTE
DENSITY OF GASES IN POUNDS PER CUBIC FOOT AT VARIOUS TEMPERATURES
DEGREES DIFFERENCE IN TEMPERATURE BETWEEN CHIMNEY GASES AND EXTERNAL AIR AT 70° F
3100
2900
2700
2500
2300
2100
1900
1700
1500
1300
1100
900
.07
.06
.05
.04
.03
.02
100
200
300
400
500
600
700
800
900
1000

static pressure diminishes towards the outlet, the total pressure also diminishes towards the outlet.

The equation representing the conditions stated above is

$$D = V + S$$

where D is the total pressure at any point in the horizontal pipe, V is the velocity pressure and S the static pressure at the same point.

While it is possible to measure the static pressure in an apparatus as sketched above, the corresponding static pressure (being a part of the total pressure) cannot be measured in a vacuum system such as we have in a chimney.

Let Figure 6 represent a chimney, containing hot gases at a uniform and constant temperature, and let D be the unbalanced pressure or draft intensity, as indicated on the draft gage, when the bottom is entirely closed by the diaphragm a. If the diaphragm is removed and there exists no frictional resistance, D will be converted entirely into velocity pressure and the static pressure as indicated on the draft gage will fall to zero.

If the bottom or the top be constricted to an area of say 2 square feet, the pressure D will act at the bottom over the two square feet of section, producing an upward flow, and the unbalanced weight of this particular column 2 feet square will be nearly neutralized, and it will be able by itself to unbalance the draft gage only slightly, but the pressure produced by the weight of the gases in the interior of the entire chimney, other than this column of 2 square feet averaged with the pressure of this column will act on the draft gage and a smaller reading will be indicated than in the first case.

If we grant that there is frictional resistance against the walls of the chimney, and the bottom is completely opened, the pressure D at the bottom will cause a flow up through the center of the chimney close to the theoretical velocity, but there will be a cylindrical shell of gases near the wall, which on account of the friction against the wall, will be more or less dormant, and the pressure produced by the weight of this shell of questionable thickness averaged with the mean pressure in the moving column will

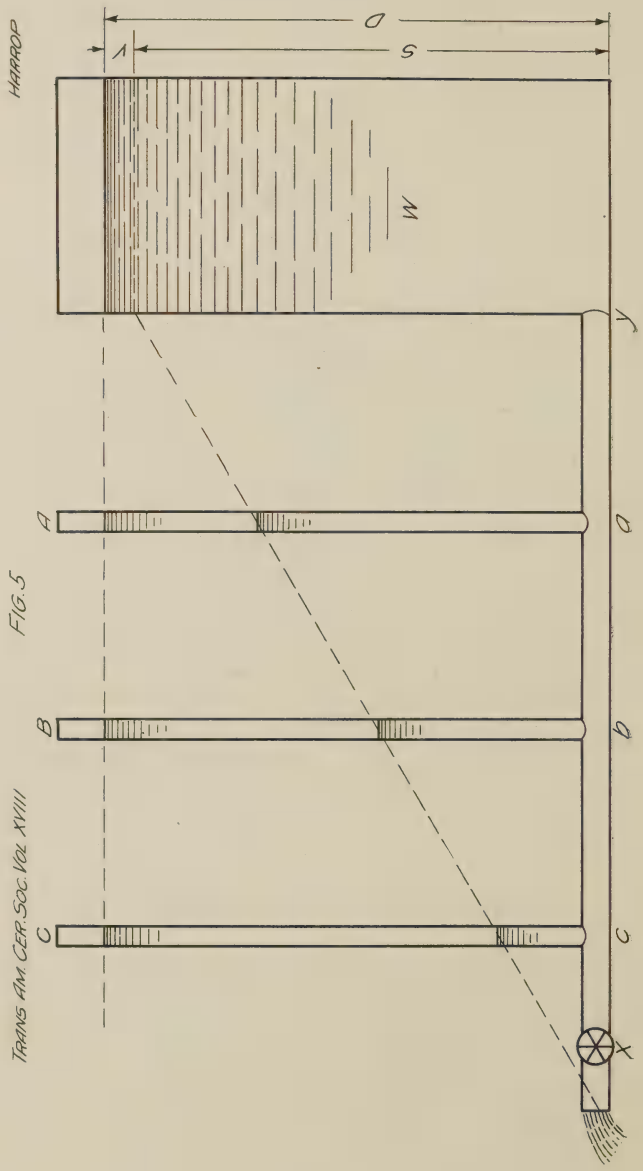


FIG. 5

TRANS AM CER SOC VOL XVIII

act on the draft gage and a much smaller unbalanced condition will be noted. It is thus seen that as the volume of gases increases, the reading of the static gage decreases and *vice versa.*

To cite a more specific case, let us assume a chimney and flue (Fig. 7) with a draft intensity of 0.6 inches at the level indicated. This means that there exists in the chimney a pressure of 0.6 inches less than atmospheric pressure, or a negative pressure. If, when the chimney is operating, the pressure tending to counteract this at the same level in the chimney are a velocity pressure of 0.2 inches and an actual static pressure of 0.3 inches (0.1 having been lost in friction), there will remain a negative pressure or

$$D \quad V \quad RS$$

draft of $0.6 - (0.2 + 0.3)$ or 0.1 inches still indicated on the draft gage. It will be noted that frictional resistance prevents the total pressure of 0.6 inches from entirely balancing this same pressure, which showed negatively when the draft intensity was observed. It is evident, therefore, that this 0.1 inch is not the real static pressure which maintains the flow, but is an uncounteracted pressure which is a measure of the frictional or other resistance. **The real static pressure, which maintains the flow, cannot be measured in an ordinary draft gage against the atmosphere, as it is produced by and is a part of atmospheric pressure.**

Our formula, therefore, covering pressures in a draft system, is the following:

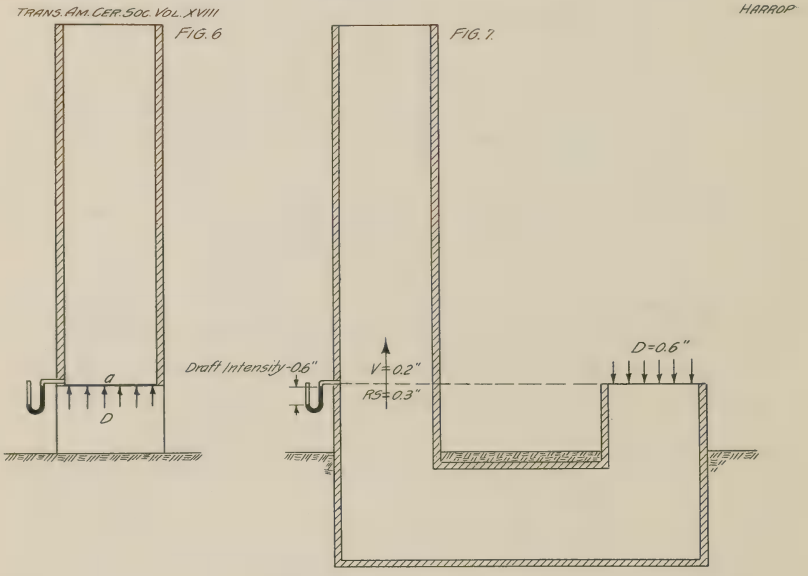
$$D = V + RS + S$$

S being the uncounteracted negative pressure at any point in the system, or the draft measured on the ordinary gage.

If the gases in the chimney in Fig. 8A are constant at temperature $T°$ and the bottom is closed at **n**, an unbalanced pressure or draft intensity, which we will indicate by the line D, will show on the draft gage. If the bottom of the chimney is opened up at **n**, and if we could measure both the velocity pressure V' and the real static pressure RS' (which is necessary to maintain the velocity V' up through the chimney), their sum will equal the intensity D as diagrammed and the draft gage will stand at zero, pro-

vided there is no frictional resistance between the opening **n** and the gage.

If, to the bottom of the chimney, Fig. 8B, is now connected a flue of equal area and with a full opening at **a,** and the pressures determined as above, we will find a smaller velocity pressure V and a still smaller real static pressure RS, the sum of which, together with the gage reading p, will also equal the intensity D. This relation is shown on the vertical line. S (the gage reading) represents the resistance in the flue up to the gage, and RS repre-



sents the resistance offered by the chimney — being smaller in this instance than shown in Fig. 8A, because the velocity is less and the resistance reduced to a still greater degree, since the resistance varies as the square of the velocity.

The volume of gases flowing will be governed by the smallest cross sectional area within the system and the accumulated frictional resistance — the actual average velocity over the cross section at any point being governed by the relative sectional area at that point, while the frictional resistance to be overcome is zero

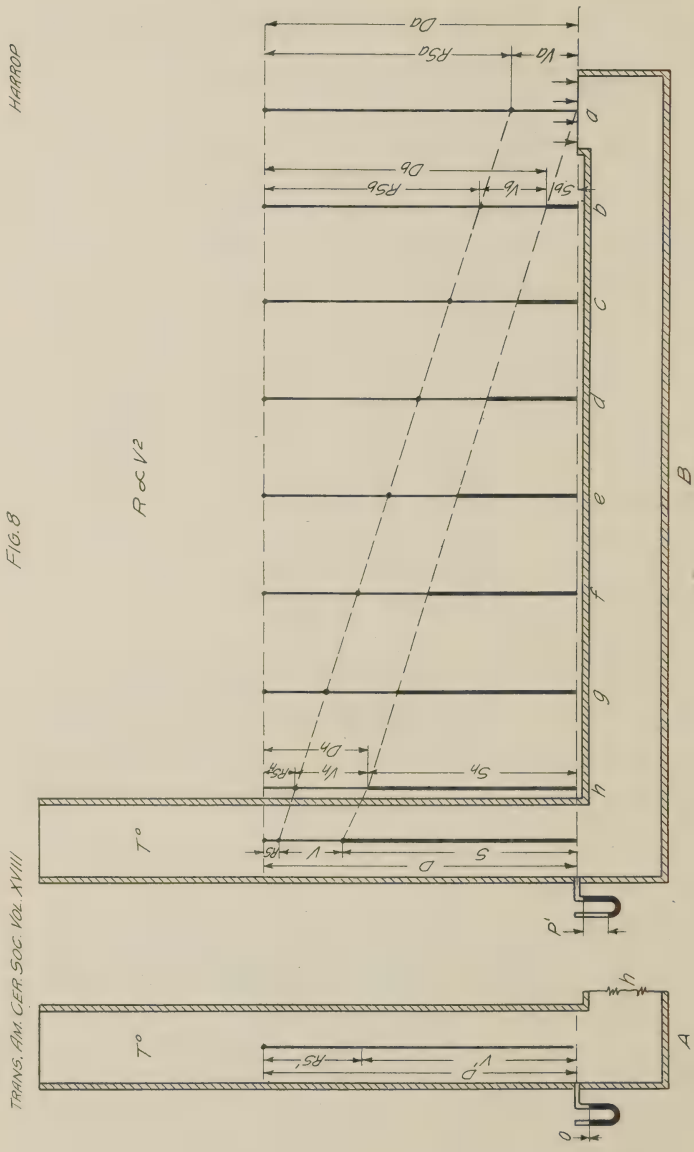
at point **a** and is cumulative through the whole system to the top of the chimney. We will find the velocity pressure, (disregarding temperature and volume changes) if reduced to the same cross sectional area, constant throughout the entire system.

If the end of the flue is closed at **a,** the draft intensity will be found to be the same at points **a, b, g** and **h.** If the flue is completely opened at **a,** the total pressure D_a will act over the entire opening and will consist of velocity pressure V_a and real static pressure RS_a, which pressures will entirely counteract the draft intensity at this point, and the draft gage will stand at zero. However, at point **b**, while the velocity pressure remains constant, the total pressure D_b and therefore the real static pressure RS_b is reduced on account of the friction encountered from points **a** to **b**, *i. e.* the total pressure at this point is unable to completely counteract the draft intensity, and this difference in pressure S_b, or the draft pressure as indicated on the gage, is a measure of the resistance up to this point. It will be noticed that this difference increases as the chimney is approached, where at point **h** the total pressure acting to produce the flow has been reduced to D_g, which at this point is largely velocity pressure. S_h represents the draft gage reading.

It is thus seen that it is possible to to have draft gage readings anywhere between zero and the maximum draft intensity, depending upon the point at which the reading is taken and the amount of resistance in the system. It also shows clearly another thing of even greater importance, namely, that the reading ordinarily obtained with a static draft gage has no direct bearing on the velocity, but instead, indicates the resistance offered to the flow of the gases.

It is often stated that the combustion of wood requires but little draft, while slack coal requires a high draft, — and these are the draft conditions which are usually found by gage readings in each case. With the above conception of the meaning of static draft gage readings, it would be much nearer the truth to say that low draft is the **result** of burning wood and high draft the **result** of burning slack coal.

Fig. 9 is a pressure diagram covering conditions in a chimney, flue and kiln. The opening **a** represents the com-


HARROP
FIG. 8
TRANS. AM. CER. SOC. VOL. XVIII
A ∝ V2
T°
T°
A
B

bined grate area of the kiln, **b** represents the resistance encountered in the kiln and **c** a damper party closed. The total pressure D_a per square foot is applied at the grates. The velocity pressure V, as indicated over the area of the chimney, is distributed over a much larger area at the grates and therefore is much less per unit of area, as indicated by V_a. The variations in the real static pressure, the velocity pressure and the static gage pressure, (indicated by the lower, heavier portion of the vertical lines) can be followed throughout the system, as in Fig. 8 B.

Draft Gage Readings in Continuous Kilns. If a draft of 0.25 inches is carried on the burning chamber of a continuous kiln and one of the cooling chambers is cut off, a certain amount of resistance will be removed and result in a drop in the static gage pressure and a slight increase in the velocity pressure.

If an additional chamber is put on ahead to water-smoke, there will result a drop in the static gage pressure and a decrease in the velocity pressure.

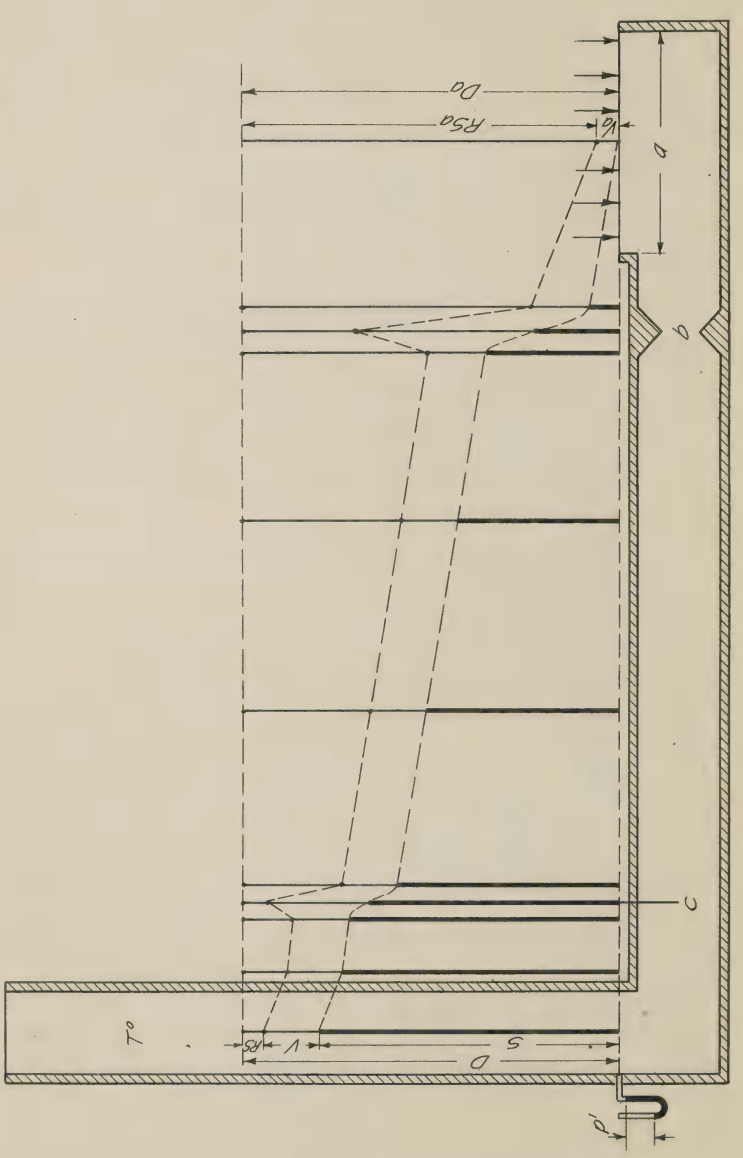
If a cooling chamber is taken off and an extra chamber is put on ahead to water-smoke, the static gage pressure will drop, and the velocity pressure will remain the same.

If the fan or chimney draft becomes weak, the static gage pressure will drop and the velocity pressure will become less. If the fan or chimney draft becomes stronger, the static gage pressure will rise, as will also the velocity pressure, although at a less rate, since the static gage pressure (and resistance) varies as the square of the velocity (cross sectional area of air passages remaining the same). This is also true of periodic kilns, where the kiln and chimney temperature increase.

Evidently no definite relation exists between the static gage pressures and the velocity of the combustion gases in a continuous kiln.

Measurement of the Velocity of Chimney Gases. If we had some means of measuring the velocity of chimney gases, we would be much more able to understand and control the rate of pressure and the velocity of the combustion gases in a continuous kiln.

An instrument known as the Pitot tube, the theory of which was explained by Bleininger in Vol. X, *Trans. Amer. Cer. Soc.*,


HARROD
FIG.9
TRANS. AM. CER. SOC. VOL. XVIII
Do
R50
16
a
b
c
T°
1/2
1
S
D
d'

is very serviceable in measuring the velocities of water, steam or high velocities of air, but the velocity of chimney gases is so comparatively low, that the ordinary variations, unless indicated on an exceedingly sensitive gage, would be hardly discernible, — the total range during the burning of a kiln, probably falling within 5/100 of an inch of water column. Another objection to the use of this instrument is the difficulty of determining the average reading, especially if the cross sectional shape of the flue is not regular.

Based upon the fact that the resistance offered to the flow of a gas varies as the square of its velocity, a comparatively recent idea is here suggested for use in measuring draft. A single static draft gage and two draft tubes are used, the latter being placed at fixed intervals in the draft system, between which the resistances are always the same. The regular connection is made between the gage and the draft tube nearest the stack, while the end of the gage usually left open to the atmosphere, is connected with the other draft tube. Thus, a differential reading is obtained on the gage. If there is no friction in the flue between the tubes, the negative static pressure will be the same at both places (if at the same level), and the gage will stand at zero. However, as there is resistance, the pressures in the two tubes will be different, and the gage will stand unbalanced, the gage reading being a direct measure of the resistance offered by the flue between the two tubes. As the velocity of the gases increases, the friction and therefore the gage reading also increases, and as the velocity of the gases decreases, there is a decrease in friction and a directly proportional decrease in the gage reading. As the velocity bears the above mentioned fixed relation to the resistance, it is a simple matter to maintain a uniform velocity or to determine the relative change in velocity or volume of gases from the change in gage reading.

ALBANY SLIP CLAY

BY ROBERT W. JONES.

A clay which will fulfill all of the many requirements of a slip is not found in any great abundance or at many localities. There are, in the United States, several localities where clay deposits have furnished, or are furnishing, material which has been successfully used as a slip for many ceramic products. Such localities are found in Texas, Michigan and in the Hudson Valley of New York State. It is probable that the material shipped from the New York localities has had a greater market and a greater production than that from any other section. At the present time the production, from the Hudson Valley, is being used to a great extent in the manufacture of electric insulators, as a bonding material in the manufacture of abrasive wheels and to some extent in the production of ornamental terra-cotta.

Lately, there has been some discussion concerning the different grades of slip-clay found in the neighborhood of Albany, New York. Some difficulty has been found in securing consecutive shipments which would give the same results in practice. In this article I am attempting to give what facts are known concerning the geological conditions of deposition of the Albany slip-clays, the chemical analyses of material from the various beds, the mineralogy and physical structure, the production and methods of mining, which facts will show the cause of these irregular shipments.

The clays of the Hudson valley are almost entirely the result of glacial action. During the retreat of the great body of ice, which at one time covered this portion of the northern hemisphere, the land stood lower than today, due very likely to the weight of the enormous ice-burden. As the ice melted back through the present Hudson valley, a "Hudson inlet" of the sea took its place reaching eventually clear through to sea-water in the St. Lawrence valley near Montreal, and thus becoming a

"Hudson-Champlain strait." Between the Hudson Highlands and Glens Falls, this strait had an expansion called "Lake Albany," and in the Champlain valley another expansion called "Lake Vermont."

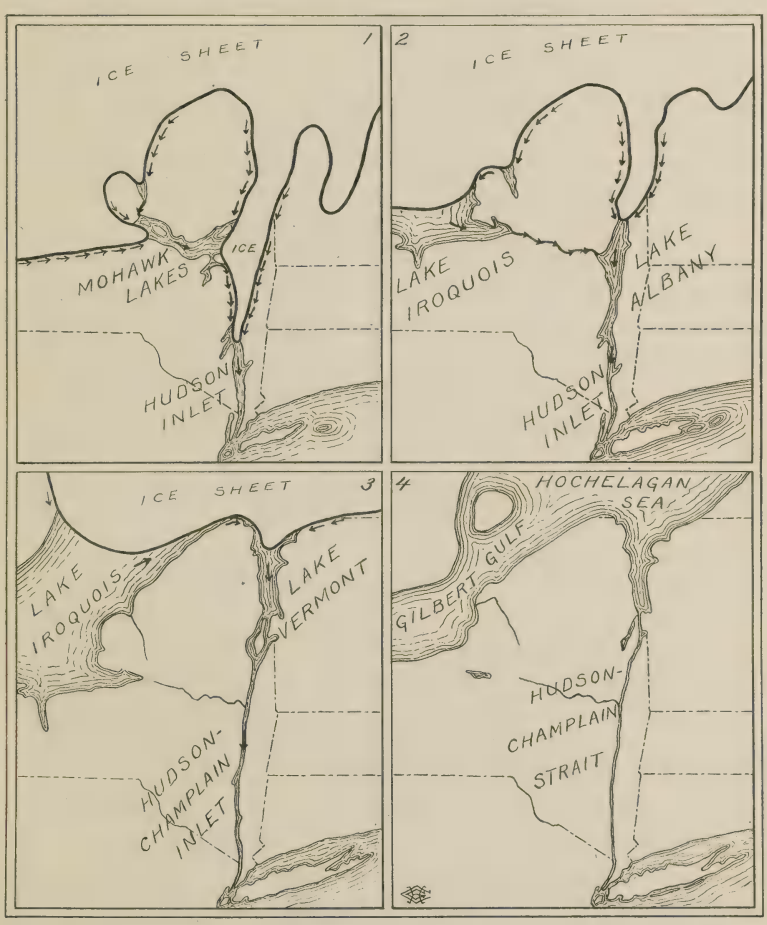


FIG. 1.

Figure 1 illustrates four stages in the retreat of the ice sheet from its terminal moraine on Long Island. The arrows show the direction of flow in the various water bodies and also of the

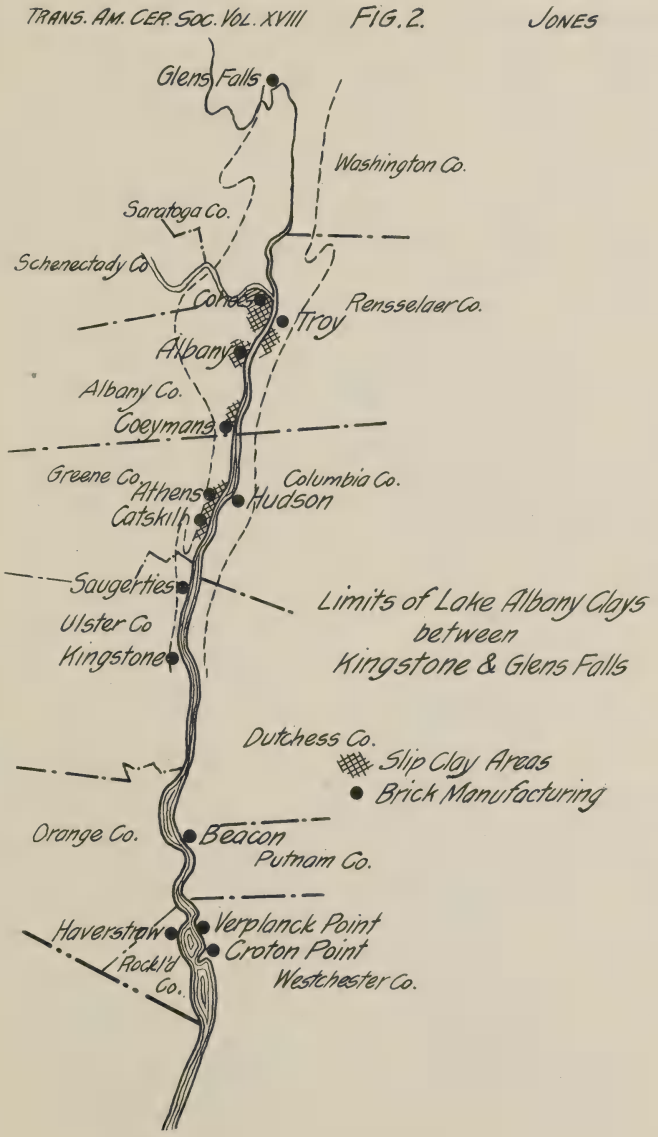
forced drainage along the ice-margin. The maps are much generalized, all unimportant details being omitted.

While these waters were of tide level, they were probably quite fresh, as is the tidal Hudson of today. The connection with the ocean through the Highlands was narrow, and there was a great inflow of fresh waters not only from the melting ice but also from the entire Great Lakes system, during the glacial blockade of the St. Lawrence outlet, which poured into Lake Albany at first through the Mohawk valley and later, around the north point of the Adirondack mountains and thence by the way of Lake Vermont.

The shore lines of this submergence may now be traced northward from New York harbor as a steadily rising plane, indicating a subsequent uplift that culminated in Canada. The amount of this uplift appears to have been over one hundred feet at Haverstraw, two hundred and twenty feet at Kingston, three hundred feet at Ravena, three hundred and fifty feet at Schenectady, four hundred and fifty feet at Glens Falls and seven hundred and forty feet at the Canadian boundary. This land rise did not wait for the complete disappearance of the glacier but followed its retreat as a wave progressing from the south to the north.

The sources of the materials for the clays and sands are to be found, in (a) the direct outwash from the ice itself; (b) fresh glacial soils left within the grasp of the forced drainage along the ice margin, or of the wave action, or of the normal drainage from the surrounding land; and (c) erosion of the bedrock of the region by these same three agencies. Deposits from these different sources naturally interfinger and overlap.

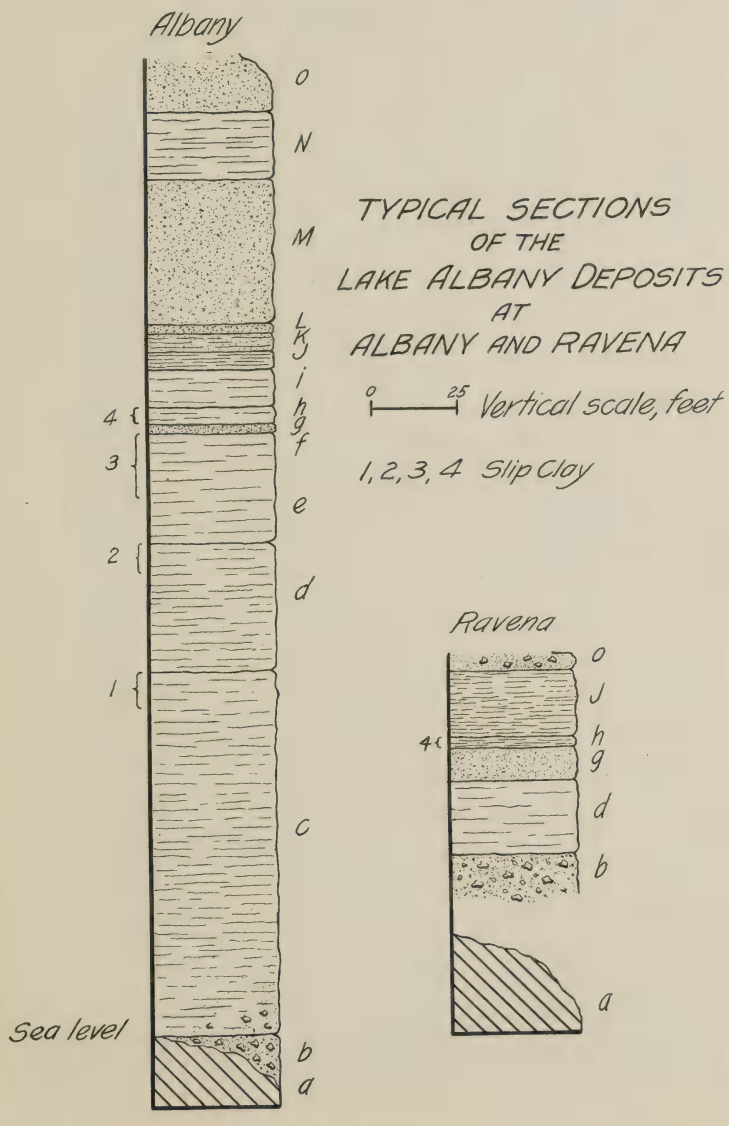
For the purpose of this article, we are only concerned with that portion of Lake Albany extending from Kingston on the south to Troy on the north. This takes in the area of the lake at points where it had its greatest width, and where the waters were, no doubt, more quiet than in other sections. This portion received sediments derived from various sources. The igneous and metamorphic rocks of the Adirondack mountains furnished a rather small amount, the siliceous rocks of the Renssèlaer plateau a slightly larger amount, the calcareous rocks of the Helder-


TRANS. AM. CER. SOC. VOL. XVIII
FIG. 2.
JONES
Glens Falls
Washington Co.
Saratoga Co.
Schenectady Co
Cohoes
Troy
Rensselaer Co.
Albany
Albany Co.
Coeymans
Greene Co.
Athens
Columbia Co.
Hudson
Catskill
Saugerties
Limits of Lake Albany Clays between Kingstone & Glens Falls
Ulster Co
Kingstone
Dutchess Co.
Slip Clay Areas
Brick Manufacturing
Orange Co.
Beacon
Putnam Co.
Haverstraw
Verplanck Point
Croton Point
Rockland Co.
Westchester Co.

berg mountains and of the Mohawk valley furnished a great portion of the bonding material, and the local dark colored shales, slates and sandstones of Normanskill age, found so abundantly throughout the Hudson valley, furnished the greatest amount of material in the form of irregular rounded grains of clear, iron-stained and opaque quartz varying in size from .0327 inch to .0003 inch in diameter. Scattered through the clays are found a few undecomposed grains of shale and sandstone. It was formerly considered that the blue and gray color of these clay deposits was derived from these occasional grains, but, after a microscopic examination of many samples, it was found that this color was due to a thin film of slightly stained calcium carbonate surrounding each quartz grain. The map (Fig. 2) gives a general outline of the clay deposits formed in this section of the lake.

The clay deposits of the Hudson valley can be divided into four groups denoting that number of eras or conditions of deposition. They are as follows: 1. glacial water sorted clays of dark red or chocolate color, such as are exposed on Staten Island; 2. the morainal blue sandy clays such as are exposed at Haverstraw, Beacon, Dutchess Junction and in the lower beds at Kingston; 3. the fine grain blue or gray clays of quiet water deposition, extending north from Kingston to Troy and included within the limits of the accompanying map; 4. the delta deposits found in the valleys of the more important tributaries, such as the present valley of the Kaaterskill creek, and at several points north of Albany (Fig. 3, N.). It is from the clays of the third group that the production of slip clay has been made. It is not possible to trace a particular bed of any of these groups for a great distance without finding a change in color, size of grain or chemical composition. This is due mainly to the great number of eastward and westward flowing tributary streams, causing a variation in deposition and deposits. As a result of this condition the deposits, especially south of Kingston, consist to a great extent of overlaping and alternating thin beds of sand and clay. To the north of Kingston this condition is not so pronounced, and the beds are fairly regular in color, composition and grain. A good example of this alternate bedding is shown in Fig 8, a photograph of an exposure of slip clay near Albany

TRANS. AM. CER. SOC. VOL. XVIII FIG. 3. JONES



During the advance and retreat of the ice-sheet, great masses of disintegrated rock were deposited throughout the valley as lateral and terminal moraines. Within the limits of the map, this deposit is always found below the clays with a thickness varying from a few inches to several hundred feet or more. One of the typical exposures of this morainal material is at North Albany, where it has a thickness greater than two hundred feet and reaches above the level of the adjacent blue clays. This North Albany deposit is well shown in Fig 4, which gives a view along a north and south line at its greatest elevation. Below the deposit of sand and gravel (Fig. 3, B.) is found the upturned beds of dark colored shales, sandstones and slates of Normanskill age (Fig. 3, A.) The morainal materials in many cases formed dams or obstructions to the flow of the current, resulting in the formation of areas of back-water where only the very finest materials were deposited. Such an area with its resulting deposits occurred in the vicinity of Albany.

After the lowering of the water, the clay deposits were left in the form of more or less dissected terraces, having in the Albany section an elevation of about two hundred feet for the top of the blue clay. Figure 5 shows these terraces as exposed along the Normanskill creek near Albany, and Fig. 6 gives a view along the upper level of this terrace west of Ravena and east of the Helderberg Mountains, which at this locality formed the western limit of the lake. Figure 6 gives some idea as to the topography of the clay deposits. This terrace has an almost unbroken extent of forty miles north and south with a width varying from one-quarter to two miles.

Typical sections of the lake deposits at Albany and Ravena are given in Fig. 3, showing the variation in a distance of about twelve miles. The bottom of the blue clay is given, for the Albany section, as being at sea-level. This is not always the case as a great many locations show a depth extending far below sea-level. As mentioned before, **(A)** and **(B)** represent the upturned Normanskill deposits and morainal material respectively. **(C)** is a bed of blue clay of varying thickness and composition. Generally this clay can only be used in the manufacture of soft-mud brick, although

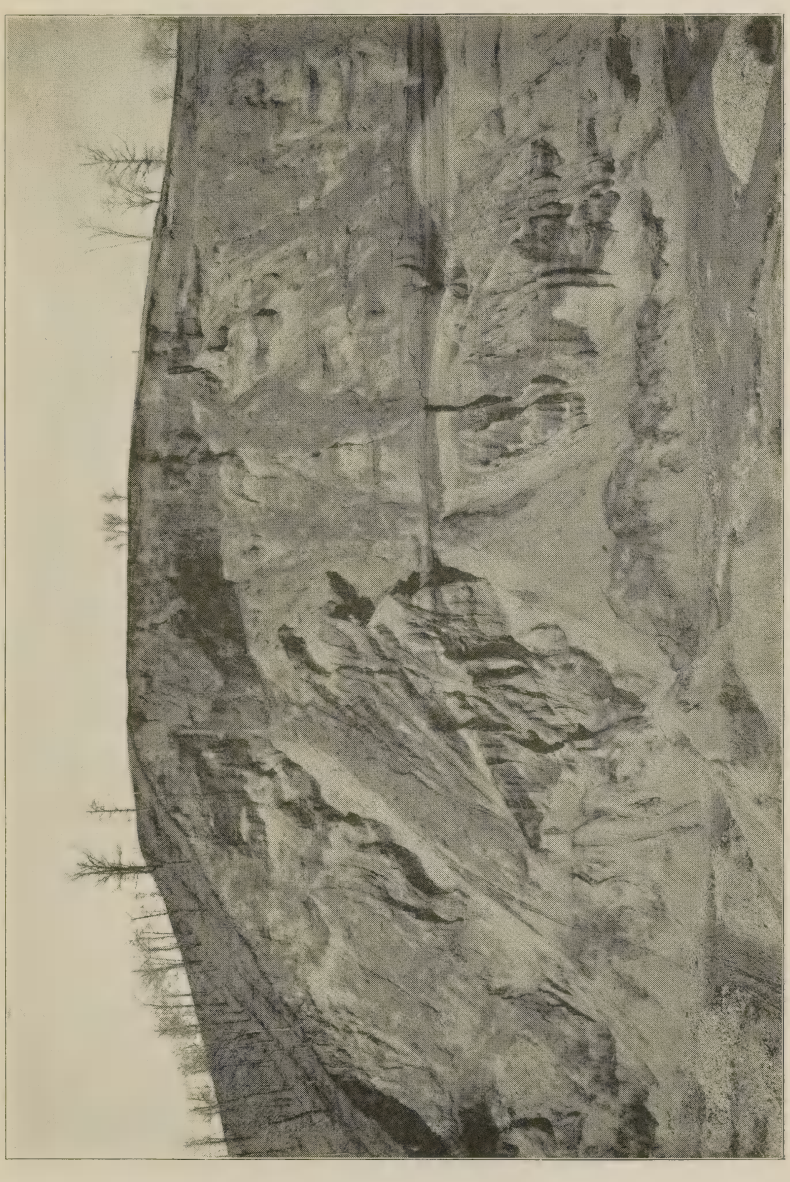


FIG. 4.

hollow blocks, common hollow brick and drain tile have been made from this material. Near the top of this division is found a bed of rather uniform clay having a thickness varying from a few feet to twenty feet. This is the lowest productive bed of slip clay. The clay from this bed can be used as a slip for certain grades of material, but for the better grades such as insulators, there has been a great amount of uncertainty as to the final result. **(D)** consists of blue clay of fine grain and more regular composition than the lower bed. This is a clay which can be used with more success in the stiff-mud machine and on the wheel for flower pots. Heavy beds of sand are absent, and in the upper portion is found a thin bed of slip clay from which was taken in the early days, the greater portion of the material for the manufacture of glazed ware which was carried on to a great extent in Albany. No shipments have been made from this bed for many years. **(E)** is a rather thin bed, sharply divided from the upper slip clay bed and resembles to a great extent the lower beds of **(D)**. **(F)** is the bed from which the principal output of slip clay is now being made. It consists of rather thick layers (about six inches) of bluish or purplish clay divided by paper-thin layers of clear quartz sand. At the one locality where this bed is exposed to the best advantage, it has a thickness of about ten feet. **(G)** consists of a two-foot layer of gray sand, and it is from this bed that considerable of the trouble comes in attempting to secure uniform shipments. **(H)** is the upper commercial slip clay bed and greatly resembles the material found in **(F)**. It has a thickness of about four feet and consists of a dark gray colored material carrying very little sand. In the bank, there is some difficulty in recognizing the division between this bed and the underlying bed of sand, and the consequence is that, with new and inexperienced labor, a considerable amount of sand will be shipped as slip clay . **(I)** consists of an eight-foot bed of highly colored blue, green and maroon clays carrying too much sand to give it any value for commercial purposes. **(J)** consists of a four-foot bed of alternating sands and clays cemented into a rather compact layer by calcium carbonate.

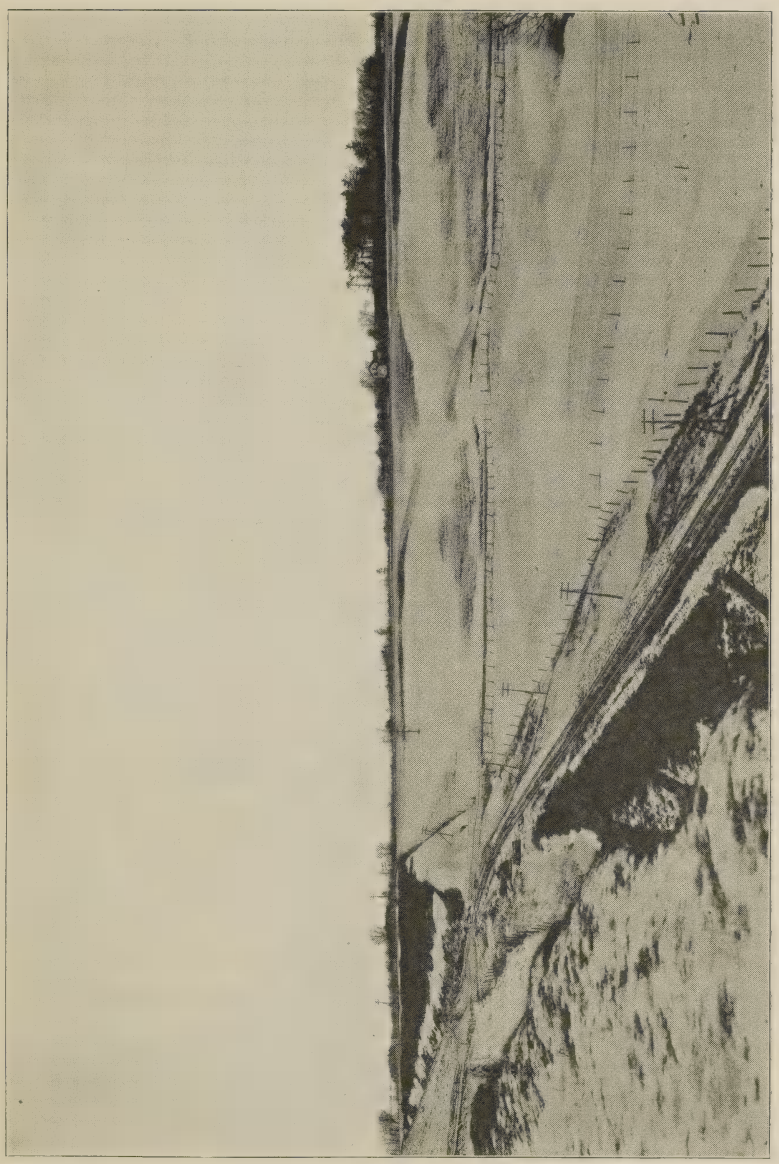


FIG. 5.

(K) is a four-foot bed of gray sandy clay uncemented. **(L)** is a two-foot layer of gray colored sand and the uppermost layer of the blue clay deposits. **(M)** consists of about eighteen feet of open, yellow, iron-stained clear quartz grains. **(N)** is a bed of brown clay alternating with thin layers of dark colored sands. This is a local delta deposit and varies in thickness up to twenty feet. **(O)** resembles **(M)** in every particular. It is in this bed near the contact with **(N)** that much of the Albany molding sand is found.

After exposure to the atmosphere and to the surface waters, the blue clays undergo a chemical change with the formation of a light or dark brown colored clay, having somewhat different physical and chemical characteristics and different working qualities especially in the manufacture of common brick. This chemical change does not seem to have had much effect on their working qualities as regards their use as a slip.

Many samples of slip clays and other Hudson Valley clays have been examined under the microscope before and after sizing, and only in one case were any minerals found with the exception of quartz, feldspar and gypsum. The great body of the clay consists of irregular rounded grains of quartz either clear, iron-stained or opaque. Some beds, such as **(L)**, will show practically nothing but clear quartz, others will have a greater proportion of iron-stained grains, and a few will be formed of the opaque variety. Some beds will be found with quartz grains varying in color according to the diameter of the grain. This is noticeable in samples taken from **(K)** where the grains measuring .0117 inch and .0081 inch in diameter consist entirely of iron-stained quartz with the rest of the sample made up of clear quartz. The feldspars are practically absent as recognizable minerals; in nearly every sample examined, there were found a few small grains but not in any case were they present to an extent where they would be noticeable without careful examination. Gypsum as a secondary mineral was fairly abundant, and, at one locality, crystals as shown in Fig. 7 were obtained in great quantities. In one sample from the slip clay beds, there was found a small perfect crystal of apatite, calcium phosphate. It is very rarely that the quartz

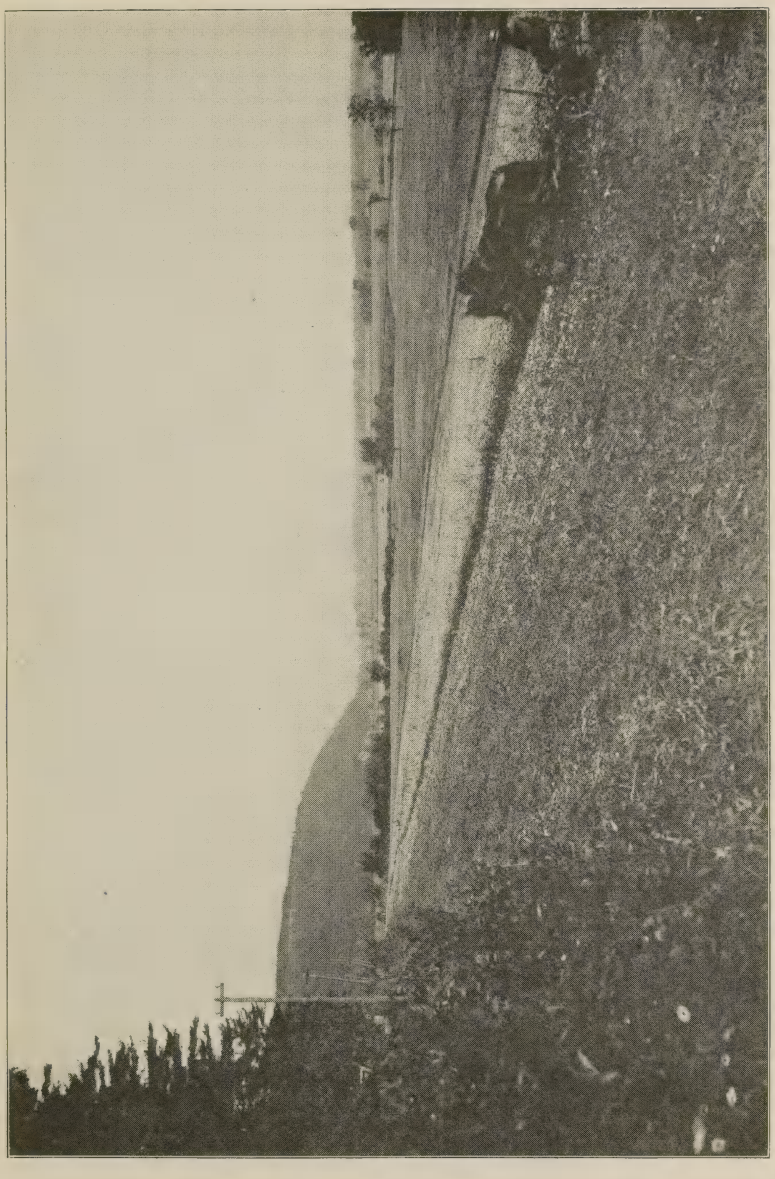


FIG. 6.

grains are free but in most cases they are cemented together into larger irregular grains by calcium carbonate or limonite. Calcium carbonate is always present as a bonding material with, also, a small amount of magnesium carbonate. There were found in many of the beds, layers of rounded sandstone pebbles derived from the adjacent rocks. It is these pebbles that account, to a great extent, for the grains having a diameter of from .0327 inch to .0081 inch inclusive.

Careful samples were taken of many slip clays and of other clays and sands for sizing tests. The samples were sized into grains having ten different average diameters varying from .0327 into to .0003 inch. In the case of the slip clays the greatest amount of material averaged .0003 inch or smaller. This formed the main body of the clay, and the average of many samples from the different slip clay beds was 83.64 percent. The next grain found in abundance was that having a diameter of .0016 inch and formed 8.68 percent of the total. The slip clay beds, both brown and blue, are remarkably uniform as to size of grain. This is shown in samples 7 to 10, inclusive, as given in Table I. The sandy layers of the slip clay beds were in most cases similar in composition to the clays, except that the grains having a diameter of from .0016 inch to .0009 inch were in greater abundance. For comparison with the slip clays there is given a blue clay, deposited in the very quiet waters of Lake Albany in what is now the valley of the Kaaterskill creek. This clay has a thickness of about sixty feet, composed entirely of quartz grains cemented by calcium carbonate, with 95.16 percent having a diameter of less than .0003 inch, only 3.90 percent having a diameter of .0009 inch and .04 percent with a diameter of .0066 inch.

There have been many chemical analyses made of the slip clays and other clays of the Hudson valley but of the entire list only those in Table II are of interest. A few are given from other localities for comparison.

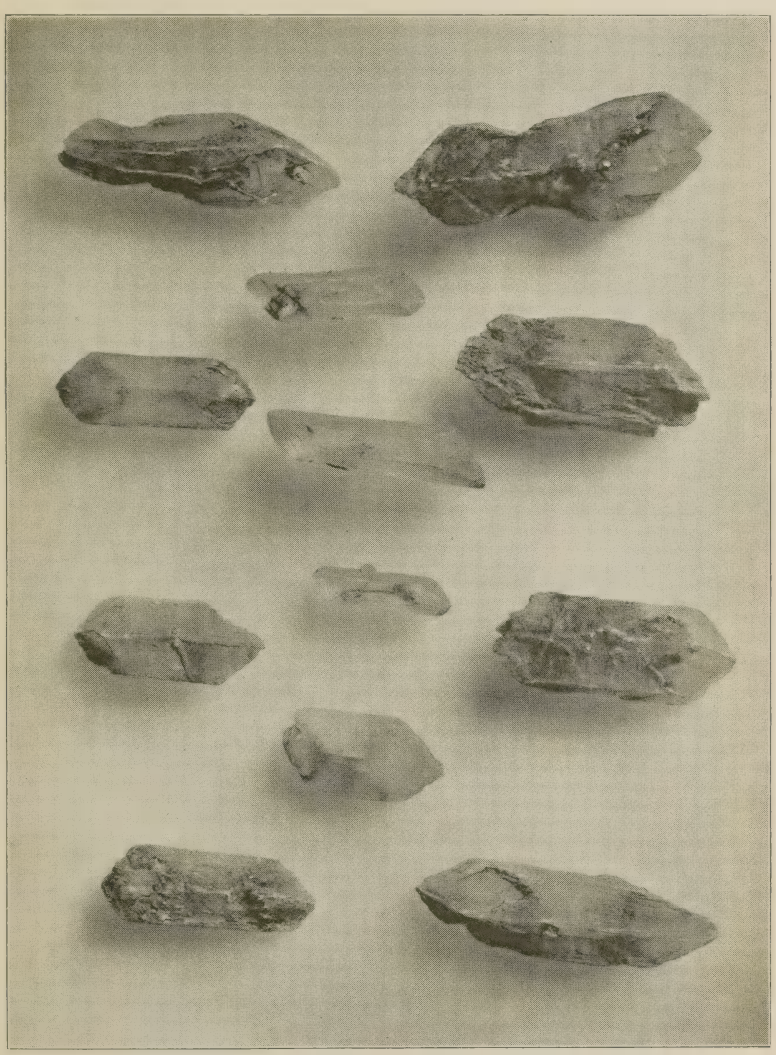


FIG. 7.

TABLE I. MECHANICAL ANALYSES OF SLIP CLAYS, SANDS AND OTHER CLAYS

SIZE OF GRAINS	No. 1	No. 2	No. 3	**No. 4**	No. 5	No. 6	No. 7	No. 8	No. 9	No. 10	No. 11
	percent	*percent*	*percent*	*percent*	*percent*	*percent*	*percent*	*percent*	*percent*	*percent*	*percent*
.0327	1.86	.....	1.20	.16	.....	.....	.10	.10	.12	.20	.....
.0163	.60	.....	.22	.32	.....	.02	.20	.20	.24	.30	.....
.0117	.56	.....	.18	.48	.....	.08	.30	.40	.24	.32	.....
.0081	.54	.....	.50	.64	.02	.04	.44	1.20	.40	.36	.04
.0072	.96	.....	.52	.72	.76	.02	.80	.80	.50	.60	.06
.0066	.40	.04	.08	.50	.50	.04	.50	.40	.50	.38	.20
.0032	6.52	.30	.44	8.00	9.56	.10	1.20	1.30	.80	1.60	.40
.0016	19.02	.60	12.48	20.00	38.96	17.64	8.00	8.40	6.00	12.00	9.00
.0009	4.66	3.90	7.10	11.00	6.76	14.00	3.00	5.40	5.00	9.00	2.80
.0003	64.88	95.16	77.28	58.18	43.44	68.06	85.46	81.80	86.20	75.26	87.50

1. Red surface clay, Kaaterskill.
2. Blue clay underlying No. 1, Kaaterskill.
3. Brown clay, Dutchess Junction.
4. Sandy layer found above the blue clays at Albany (Fig. 3, L.)
5. Yellow sand below blue clays, Hudson.
6. Sandy layer in the slip clay beds, Albany (Fig. 3, G.)
7. Blue slip clay, Rensselaer (Fig. 3, E.)
8. Blue slip clay, Rensselaer (Fig. 3, F.)
9. Brown slip clay, Rensselaer (Fig. 3, H.)
10. Brown slip clay, South Troy (Fig. 3, F.)
11. Blue slip clay, South Troy (Fig. 3, E.)

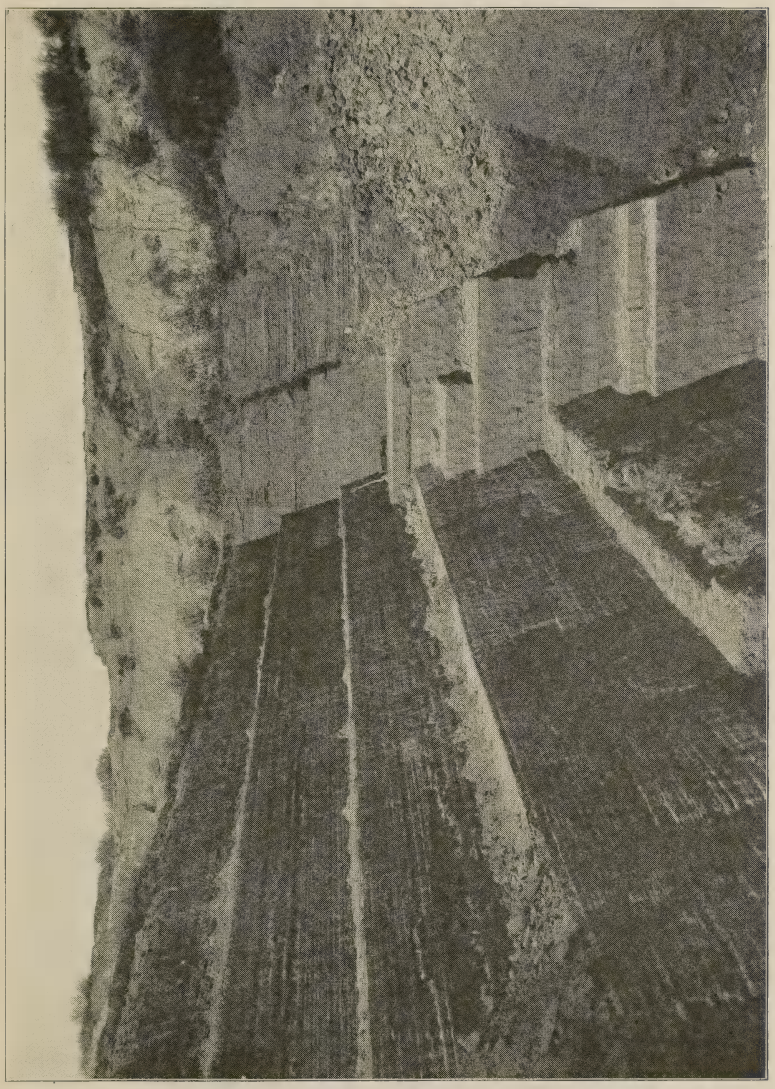


FIG. 8.

TABLE II. CHEMICAL ANALYSES.

	No. 1	No. 2	No. 3	No. 4	No. 5	No. 6	No. 7	No. 8	No. 9	No. 10	No. 11
	percent	*percent*	*percent*	*percent*	*percent*	*percent*	*percent*	*percent*	*percent*	*percent*	*percent*
SiO_2 (combined)	12.85	15.65	12.04	53.34	54.72	50.48	54.88	50.58	56.08	56.44	60.56
SiO_2 (free)	31.09	47 98	30.20								
Al_2O_3	11.17	13.57	11.08	22.27	15.63	22.85	17.96	13.15	11.50	18.74	16.08
Fe_2O_3	3.81	7.77	5.07	4.40	5.22	8.40	9.40	6.00	11.37	5.11	4.53
CaO	11.64	2.55	15.99	5.64	7.20	3.68	5.12	5.92	5.75	6.28	5.76
MgO	4.70	1.47	6.36	2.88	2.76	1.46	1.30	3.09	2.90	2.90	2.84
TiO_2	n. d.	n. d.	n. d.	1.00	.90	.95	1.00	1.05	.80	.95	.95
K_2O	3.61	2.63	2.68	3.31	3.19	.81	3.01	3.78	1.11	1.08	2.35
Na_2O				1.27	1.47	3.72	1.52	1.26	3.40	2.07	1.84
H_2O (combined)	3.90	4.75	4.58	.....	.....	.....	.....	.....	.....	.....	.....
H_2O (free)	15.63	2.90	12.00	.....	.....	.....	.....	.....	.....	.....	.....
Moisture	.....	.....	.....	1.60	1.16	1.42	1.64	1.36	1.50	.83	.63
Ignition	.....	.....	.....	8.34	7.90	7.94	8.22	8.80	8.32	8.47	6.44
CO_2	n. d.	n. d.	n. d.	n. d.	n. d.	n. d.	n. d.	n. d.	n. d.	n. d.	n. d.
SO_3	n. d.	n. d.	n. d.	n. d.	n. d.	n. d.	n. d.	n. d.	n. d.	n. d.	n. d.

n. d. = not determined.

TABLE II — Concluded.

	No. 12	No. 13	No. 14	No. 15	No. 16	No. 17	No. 18	No. 19	No. 20	No. 21	No. 22	No. 23
	percent	*percent*	*percent*	*percent*	*percent*	*percent*	*percent*	*percent*	*percent*	*percent*	*percent*	*percent*
SiO_2 (combined)	53.68	58.24	55.44	58.92	57.16	57.82	60.19	52.70	58.15	58.50	59.48	14.33
SiO_2 (free)												46.26
Al_2O_3	16.26	17.80	14.74	12.06	20.07	14.98	2.63	21.48	24.20	18.95	11.54	12.46
Fe_2O_3	4.52	5.20	4.99	4.00	4.87	4.26	1.04	7.02	5.65	7.35	4.13	5.79
CaO	5.76	4.72	5.16	6.15	3.51	4.68	10.90	3.49	2.25	2.11	6.28	6.84
MgO	3.69	2.99	5.44	1.97	1.54	2.28	3.90	.43	.24	2.00	3.35	3.28
TiO_2	.90	.80	1.10	n. d.	n. d.	n. d.	n. d.	n. d.	n. d.	.....	.90	.....
K_2O	4.29	3.32	1.35	3.01	1.24	3.26	.73	1.45	.75	2.93	n. d.	4.39
Na_2O		1.42	3.64	1.71	2.11	1.90	.41	.82	.20			
H_2O (combined	.....	.....	.....	2.02	2.72	2.11	4.47	.....	.....	.....	.....	4.36
								7.46	6.95			
H_2O (free)	.....	.....	.....	1.89	1.17	1.76	2.11	.....	.....	.....	.....	1.43
Moisture	1.48	1.00	1.08	.....	.....	.....	.....	.....	.....	.....	.48	.....
Ignition	8.12	7.70	7.64	.....	.....	.....	.....	.....	.....	.....	7.88	.....
CO_2	n. d.	n. d.	n. d.	7.01	4.18	6.16	13.13	3.21	2.05	n. d.	n. d.	n. d.
SO_3	n. d.	n. d.	n. d.	.42	1.06	.29	.....	.82	.32	n. d.	n. d.	n. d.

n. d. = not determined.

1. Slip clay, Rowley, Michigan, Ohio Geological Survey.
2. Slip clay, Brimfield, Ohio, Ohio Geological Survey.
3. Slip clay, Sharonville, Ohio, Ohio Geological Survey.
4. Slip clay, Rensselaer, N Y., (Fig. 3, (3) upper), Norton Co.
5. Slip clay, Rensselaer, N. Y., (Fig. 3, (3) lower), Norton Co.
6. Slip clay, Rensselaer, N. Y., (Fig. 3, (3) lower), Norton Co.
7. Slip clay, Rensselaer, N. Y., (Fig. 3, (3) lower), Norton Co.
8. Slip clay, Rensselaer, N. Y., (Fig. 3, (3) upper), Norton Co.
9. *Slip clay, Rensselaer, N. Y., (Fig. 3, (3) lower), Norton Co.
10. *Slip clay, Rensselaer, N. Y., (Fig. 3, (3) lower), Norton Co.
11. *Slip clay, Rensselaer, N. Y., (Fig. 3, (3) lower), Norton Co.
12. *Slip clay, Albany, N. Y., (Fig. 3, (3) lower), Norton Co.
13. *Slip clay, Rensselaer, N. Y., (Fig. 3, (3) upper), Norton Co.
14. *Slip clay, Rensselaer, N. Y., (Fig. 3, (3) upper), Norton Co.
15. *Slip clay, Albany, N. Y., (Fig. 3, (3) upper), R. W. Jones.
16. *Slip clay, Albany, N. Y., (Fig. 3, (1), R. W. Jones.
17. *Slip clay, Albany, N. Y., (Fig. 3, (2), R. W. Jones.
18. Gray sand, Albany, N. Y., (Fig. 3, (L), R. W. Jones.
19. *Slip clay, Catskill, N. Y., E. Tuceda.
20. Slip clay, Catskill, N. Y., E. Tuceda.
21. *Slip clay, Catskill, N. Y., R. W. Jones.
22. *Slip clay, Albany, N. Y., Norton Co.
23. *Slip clay, Albany, N. Y., Ohio Geological Survey.

* Indicates blue slip clays from the New York localities.

I have given these analyses mainly for the reason that, as far as I know, it is the first time that anything like a fairly complete list of the Hudson river clay analyses has been available. It is impossible to get much information from the chemical analyses of either the slip or brick clays as to their behavior during fusion. Generally the blue slip clay will be higher in SiO_2, with about 56.75 percent as compared with 52.70 percent for the brick clays; 15.47 percent Al_2O_3 in the slip compared with 21.48 percent in the brick; 5.73 percent Fe_2O_3 compared with 7.02 percent; 5.78 percent CaO compared with 6.23 percent; 3.23 percent MgO compared with .90 percent; alkalies 3.25 percent compared with 2.27 percent. The brown clays will average about 53.34 percent SiO_2 compared with about 58.15 percent for the brick clay; 20.65 percent Al_2O_3 compared with 24.20 percent; 4.73 percent Fe_2O_3 compared with 5.65 percent; 5.84 percent CaO compared with 4.02 percent; 2.98 percent MgO compared with .52 percent; alkalies 5.50 percent compared with .95 per-

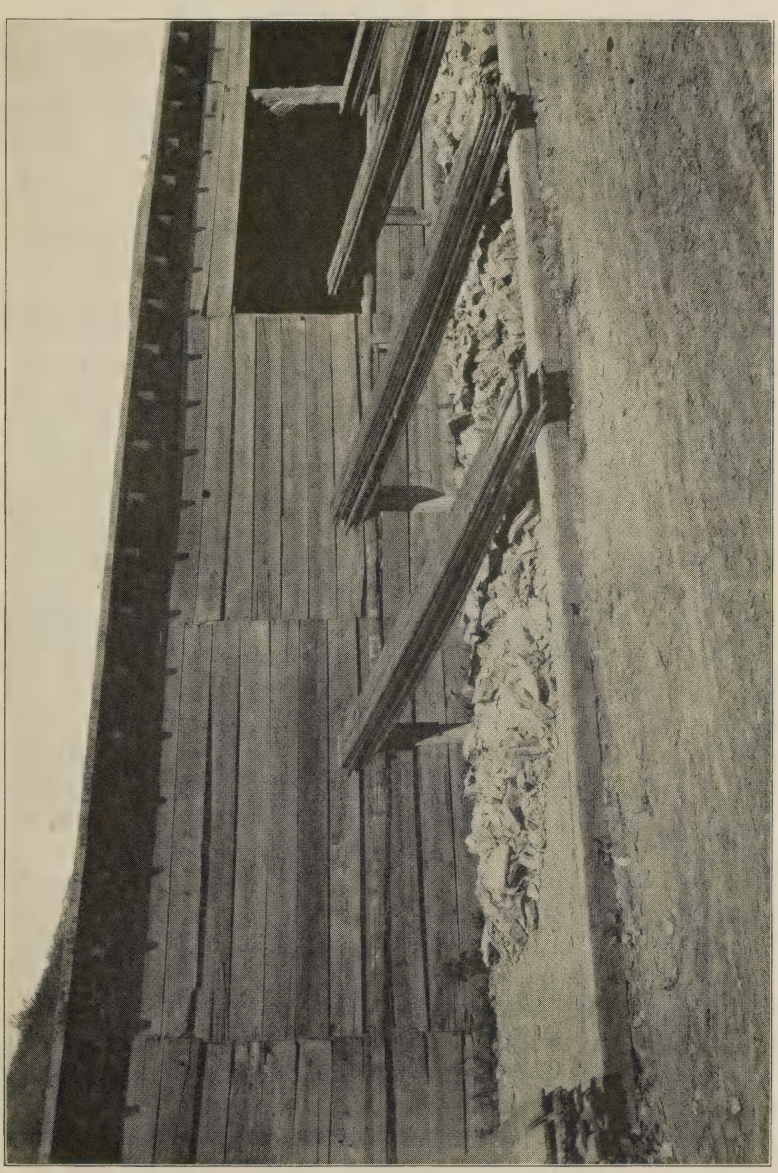


FIG. 9.

cent. These are averages from a great many analyses and are not to be depended upon to give any definite idea as to the comparative composition of the various clays.

It is almost impossible to give any estimate as to the past or present output of the slip clays in New York State. In the early days no records were kept, and at present, due to the small number of producers, it is impossible to give the figures without giving confidential information. No attempt has been made by any person or corporation to open a slip clay bank on a large scale, and no producer, in the slip clay business exclusively, is making any attempt to dry clay by artificial means. The method of working, by benches, is well shown in Fig. 8. The upper layers are carefully removed by spading, until the slip clay beds are reached. The slip clay is then cut into squares by means of a wide spade, shoveled into carts, dried by exposure to the sun and stored in bins until ready for shipment. An examination of Fig. 8, will give a good idea as to the alternate bedding of clays and sands as they appear in openings made in the Hudson river clays. The lower beds correspond to **(F)** of Fig. 3. The lower bench along the face of the opening corresponds to **(G)** Fig. 3. The uppermost layer in the face of the opening corresponds to **(L)** Fig. 3. Above these clays and across the face of the illustration are found the open yellow sands corresponding to **(M)** Fig. 3. Fig. 9 shows the method of drying. The clay is laid on the ground and during wet weather is covered with boards.

I am greatly indebted to Professor George H. Chadwick, University of Rochester, for assistance in the preparation of Fig. 1, and for other information regarding the geology of the clays, and to Mr. Ross C. Purdy, Worcester, Mass., for assistance with the chemical analyses.

THE THEORY OF FLOTATION AND ITS RELATION TO THE DRESSING OF CLAYS

BY THOMAS M. BAINS, JR.

Flotation is the process of separating substances by the utilization of certain physical or chemical properties, which causes one or more of the substances to float, while the remainder sink in the solution in which the substances are held. This process is generally the reverse of the ordinary methods of ore-dressing, which utilizes the specific gravity of the substances to effect the separation. Thus in separating galena, (specific gravity about 7.5) from quartz (specific gravity of 2.5) by flotation, the heavier mineral floats, while the quartz sinks to the bottom.

Like all great discoveries, the general phenomena connected with flotation have been known for ages. Hero of Alexander, made a steam toy before the Christian era, embodying the concept of reaction; and Branca in 1629 described the turning of a wheel by the impulse effect of a jet of steam from a nozzle. But the modern turbine era did not begin till the invention of the De Laval turbine in 1883. The principles of static electricity were studied centuries ago, but it was not until the nineteenth century that any practical uses were found for the same. So it has been with flotation.

The housewife sees flotation phenomena every day. When she places powdered cocoa in a cup and adds boiling water, the cocoa immediately becomes wetted and forms first a paste and then a solution. No foam or froth will remain on the surface. If, however, cold water, or better, cold cream be added, the cocoa refuses to form a paste, till pressure with the spoon is exerted. Then if boiling water is added to the paste, a foaming liquid results with a very persistent froth. In whipping cream, it is better to have the cream and utensils cold, or the cream may refuse to whip. The cream whip now on the market, to aid the formation of the froth, is a flotation reagent. As we sit and watch the bub-

bles in our beverages that effervesce, one of the phenomena of flotation is presenting itself. The laundress may wash dirty clothes with soap, but in cold water she will never get the clothes white. However, if the clothes be boiled, they become spotlessly white. If the same dirty clothes were dragged through salt water in the wake of a ship, the bubbles from the propellers would cleanse the clothes, and even in cold water, the same whiteness would be obtained as with boiling water.

Many mining people lately have been trying to give Mrs. Carrie Everson the credit for the discovery of flotation as applied to the recovery of sulphides from their gangue. Although her patents deal with sulphides, oils, acids and gangue, still if one tried to separate, commercially, sulphides by the descriptions in her patents, failure would result. The flotation of today has not one-hundredth as much resemblance to the Everson patents, as the Cutis turbine has to the engine of Hero.

It has been known for a long time that some minerals have a great affinity for grease or oils. This was first successfully used by Elmore, who mixed the ore with a large quantity of oil, several times the weight of the ore frequently, and the sulphides were collected in the oil, while the gangue sank to the bottom. This process necessitated a large quantity of oil and the cost was prohibitive. While using a bulk flotation process like this at the Broken Hill mill, in Australia, certain men reduced the quantity of oil added, and when ten percent oil was added, flotation ceased and coagules of oil and sulphides formed and hung suspended in the liquid, or else settled lightly on the gangue. On decreasing the percent of oil still further, — below about one and one-half percent, — it was noticed that a froth formed consisting of sulphides and bubbles with thin films of oil. This was really the commencement of a practical flotation method. Today, less than one percent of oil is used, and even flotation without oil is often successful. While this side was being developed, the surface tension phenomena was being used to produce flotation. It was found that certain finely divided minerals were hard to "wet," while others wetted readily and sank, when placed on the surface of a liquid, like water. This process is used today in the Wood flotation machine.

Another side of flotation was being developed at this time. It was found that any gas bubbles liberated in solution would collect the sulphides and bring them to the surface. If carbonates in the ore were acted upon by acids, CO_2 bubbles acted as carriers. However, without oil, the bubbles broke on reaching the surface and their burdens of sulphides dropped back toward the bottom again. The very finely divided particles, however, would float, due to surface tension phenomenon.

Various theories have been advanced to explain flotation, but the one that seems to cover the ground most thoroughly is the electrical theory. Air blown through water, or beaten up in it, is ionized, equal plus and minus ions resulting. The rate of diffusion in water, however of the positive ion is greater than that of the negative, thus the air becomes negatively charged. Air forced through canvas will become electrified, due to frictional causes. These charges are not presistent, however, unless the bubble has an insulating film about it. A charged stick of sealing wax is immediately discharged when immersed in water, but if coated with oil or vaseline, the charge will remain for some time. The charge on the air bubbles may be made to remain by:

First, by surrounding the bubble with a non-conducting film of some insulating material, and second, by the addition of certain substances, which may be divided into the two following classes:

(a) Colloids, which add charges of one sign only to the solution — these charges appear to attract the opposite kind of charge from the bubble and keep it from being discharged;

(b) Substances, like alcohol, that have practically no ionization in water and are supposed to consist of large molecules, giving a small percentage of ions in solution which would mean a slower dissipation of the charge.

Pure water splashed against a plate causes the water to become negatively charged and the air positively charged. Weak salt solution gives the opposite charges to the air and the water. Certain chemicals increase or decrease or even reverse the charges. Rosaniline increases the normal effect, giving a stronger charge to the air particles if minus. Strong oxidizing agents, as potas-

sium permanganate, tend to reverse the normal effects. Reducing agents seem to increase the normal effects.

In ordinary froth flotation, the air bubbles are charged either by beating of the air into the water or by forcing air through a medium like canvas or a certain kind of tile. This forms an immense number of small bubbles. The oil added is generally negatively charged, the sulphides positively charged. The oil is attracted and surrounds the sulphide, and the bubble attracts the sulphide, if the film of negatively charged oil is not too thick. When over a couple of percent of oil is used, the film is too thick and the repulsion of the negatively charged oil and bubble is greater than the attraction of the bubble for the sulphide. Thus coagules or balls of sulphides and oil form, that do not float. When enough oil is added so that the specific gravity of these coagules become less than that of the solution, flotation again takes place.

The two types of frothing machines, commercially, are the Mineral Separation machine, using beaters to form the bubbles, and the Callow cells which uses the canvas or other permeable bottom, through which air is forced at two to five pounds pressure. The one great point about froth flotation with these machines is, that there shall be a great number of electrically charged bubbles formed during the time of operation. In both cases acids, sulphuric generally, are used in a great majority of separations. The acid seems to cause the gangue to remain wetted, and the concentrate is much cleaner. In many cases, the flotation of certain sulphides require acid, no flotation occurring until the pulp is acidified. The heavier oils, such as pine tar or crude oils, give a more persistent froth, while the lighter oils increase the solubility of these heavy oils, decrease the surface tension of the solution, so that a greater number of smaller bubbles are formed with the other conditions remaining the same. Pure water cannot be beaten into a good froth, but if a drop of eucalyptus oil be added, all the water can be turned to froth. Turpentine and some other substances also seem to act in a way that would indicate that the charges on the bubbles are increased by these substances.

Other minerals beside the sulphides are found amenable to flotation processes. Graphite in clays may be separated by flota-

tion. Pyrite, molybenite, metallic particles, like native gold and copper may be floated.

It is possible by preferential flotation to separate one floatable mineral from another one. For instance, zinc blende and galena both readily float. By special methods the galena can be floated and the blende wetted. Then the blende can be recovered later by flotation. Minerals of the same specific gravities may be separated — this being, of course, impossible with gravity separation. The fineness of the crushing for flotation varies. Minerals as coarse as 20 mesh are often floated, while the common practice is to crush to 100 mesh or finer. The finer the particles the more water is necessary to be used in the mill pulp, and the harder it is to get a clean concentrate.

A couple of examples of the percentages of oils used in practice may be interesting:

Inspiration Copper Co. —

80 percent crude coal tar-collector,
20 percent coal tar creosote-frothing agent,
40 percent crude coal tar-collector,

Daly-Judge Mining Co. —

40 percent crude coal tar-collector,
40 percent creosote } frothing
20 percent pine oil }
1 to 1½ pounds per ton of ore or 0.05 to 1 percent pounds.

NOTES ON THE MANUFACTURE OF PORCELAIN PYROMETER TUBES[1]

BY W. L. HOWAT

Owing to the curtailment of the supply of Marquardt pyrometer tubes, manufactured by the Royal Porcelain Works, Berlin, an attempt to manufacture tubes having similar properties for use in their laboratories was made by the Bureau of Standards.

An analysis of a Marquardt protection tube from which the glaze had been sandblasted was as follows:

SiO_2	35.12 percent
Al_2O_3	63.20 percent
K_2O	1.65 percent

With the above analysis as a guide, a body mixture having a composition as follows was prepared:

Body		Grcg No. 1	
	percent	*percent*	
Grog No. 1	45.7	70.0	Calcined Al_2O_3
Grog No. 2	7.3	22.0	North Carolina kaolin
North Carolina kaolin	17.0	8.0	Potash spar
Florida kaolin	5.0		
Tennessee ball clay	15.0	**Grog No. 2**	
English china clay	10.0	64.0	Potash spar
		36.0	Calcined Al_2O_3
	100.0		

Preparation of Grogs and Body. After thorough mixing dry each grog was calcined to cone 19 and crushed to pass a 120 mesh sieve. At cone 19, grog No. 2 was vitrified and partly fused, and grog No. 1 was sintered. The body was wet ground three hours in a ball mill and filter pressed.

[1] By permission of the Director, Bureau of Standards.

Manufacture of Tubes. The first tubes were molded in the stiff-mud condition on a horizontal piston plunger machine having a die $^{13}/_{16}$ in. in diameter. The tubes were cut into 24 in. lengths, sealed at one end, and the regulation collar cemented to the other end. Considerable difficulty was experienced in the drying of the machine-made tubes on account of the tendency to warp. This warping was doubtless due, in part, to faulty die construction and unevenness of pressure. The warping was increased by burning.

More satisfactory results were secured by casting the tubes in plaster molds. One-piece plaster molds having holes ¾ in. in diameter and 30 in. in depth were prepared.

The filter pressed body was blunged in a ball mill with water and a predetermined amount of sodium carbonate and sodium silicate solution, to give the desired consistency for casting. The specific gravity of the slip was 1.874, the viscosity being such that the time required to run 200 cc. from the flow viscosimeter was 2 minutes.

In casting the above slip no difficulties were experienced in producing tubes having walls and ends of uniform thickness. The tubes released easily from the molds in from 20 to 30 minutes after casting. The tubes were placed horizontally on a glass plate during drying. The drying behavior of the cast tubes was very satisfactory, there being no difficulties from warping or cracking.

Burning. The tubes were biscuited to cone 05, being suspended vertically through holes in the lids of special saggers made for the purpose. A majority of the tubes were in good condition when taken from the biscuit kiln. A few of the tubes were warped, although it was found later that this was due to the lack of uniform bearing of the collars of the tubes on the sagger.

A glaze having the following composition was applied to the tubes by spraying, and the glazed tubes were placed vertically in the saggers as before and fired to cone 17.

Glaze.

$$\left.\begin{matrix} 0.15\ K_2O \\ 0.20\ MgO \\ 0.65\ CaO \end{matrix}\right\} 1.0\ Al_2O_3 : 10.0\ SiO_2$$

At cone 17 a good bright glaze is produced which, however, is somewhat low in refractoriness as evidenced by sticking when used at temperatures above 1350° C. although very satisfactory at lower temperatures.

Conclusions. The tubes are being used in the laboratory kilns of the Bureau and are giving very satisfactory results. At a temperature of 1450° C., the resistance to deformation has been found to correspond favorably to that of the Marquardt tubes. The resistance to temperature changes appears satisfactory and is not exceeded by that of the imported tubes.

The use of a more refractory glaze, or one which softens at a higher temperature, would improve the quality of the tubes, and glazes of this type have been prepared and are being tested.

DISSOCIATION OF CALCIUM SULPHATE AND THE REMOVAL OF DRYER SCUM

BY A. E. WILLIAMS

Dryer scum on clay products has often been removed by maintaining strong reducing conditions in a kiln at the maximum temperature of burning. This is because calcium sulphate may be more easily dissociated in a reducing atmosphere than in an oxidizing atmosphere. The disadvantage of this practice is that the color of the clay is permanently darkened, and red colors can not be obtained.

The following work was done to determine the minimum temperature at which calcium sulphate can be dissociated in contact with clay, under heavy reducing conditions. If this temperature proved low enough, dryer scum might possibly be removed by carrying on heavy reducing conditions at a temperature where no permanent change in color of the ware would be produced. Experiments to determine the approximate temperature of dissociation of calcium sulphate under reducing conditions were carried on in the following manner:

A porcelain combustion boat containing the sample was placed in a quartz combustion tube one inch in diameter, and heated in an electric furnace. A mixture of CO and CO_2 gas was prepared by decomposition of oxalic acid by sulphuric acid, dried over H_2SO_4 and P_2O_5 and drawn thru the tube by suction. When CO alone was used, the CO_2 was absorbed by bubbling thru a KOH solution. The rate of flow of the gas was kept approximately constant. To avoid air leakage, all connections were closed with sealing wax. The furnace was heated at the rate of 400°C. per hour, and air was drawn thru the tube until 450°C. was reached when the gas was admitted.

The following is a description of the experiments run, and the results obtained.

Experiment 1:—One-half gram of powdered gypsum was heated to 850°C. in an atmosphere of CO. At this temperature a flame showed in the tube for a short time, evidently oxidation of CO by the sulphate. The temperature increased at this point quite rapidly to 890°C. and continued at this point for one-half hour, at which time the experiment was terminated. The resultant product showed a loss in weight of only 7.6 percent and a strong test for SO_3 was obtained showing complete decomposition had not taken place. The presence of the flame would indicate, however, that some oxidation was going on.

Experiment 2:—In this experiment the conditions of number one were reproduced, except the temperature of 890°C. was maintained for one hour. As a result, a strong test for H_2S was obtained from the sample, showing a distinct decomposition of the sulphate. Complete dissociation had not been obtained, however, as a test for SO_3 was also given.

Experiment 3:—One-half gram of a mixture of 20 percent gypsum and 80 percent clay was heated to 800°C. and held for one hour at this temperature in an atmosphere of CO. Partial decomposition had taken place. The same tests were given as in Experiment 2.

Experiment 4:—Experiment 2 was repeated extending the time to two hours at 800°C. This sample showed no evidences of SO_3 remaining and gave a strong test for H_2S. Evidently complete decomposition.

Experiment 5:—In this experiment the same mixture was used as in Experiments 3 and 4. A mixture of CO and CO_2 was was used, however, in place of pure CO. The proportion of CO to CO_2 was that produced by the decomposition of oxalic acid by H_2SO_4. This gas analyzed with an Orsat apparatus gave 44 percent CO_2, 35 percent CO and 2 percent O. The sample was held in this atmosphere for two hours at 800°C. Complete decomposition of the sulphate was produced as in Experiment 4.

Experiment 6:—Conditions of Experiment 5 repeated, except a maximum temperature of 750°C. was maintained for two hours. Slight decomposition was produced, but a strong test for SO_3 was obtained.

Experiment 7: — Sample treated as in Experiment 5, except held at 700°C. for one hour. No decomposition of the sulphate evident.

Experiment 8: — Same as Experiment 5, except sample was held at 700°C. for two hours. No decomposition of sulphate evident.

Experiment 9: — Same as Experiment 5, except sample was held at 700° C. for three hours. A slight test for H_2S was obtained and a strong test for SO_3. Slight decomposition.

Experiment 10: — As silica and Fe_2O_3 are reported to lower the temperature of dissociation of $CaSO_4$, a mixture of 39 percent Fe_2O_3, 28 percent $CaSO_4$, and 33 percent sodium silicate was heated in an atmosphere of CO. The temperature was raised to 890°C. and held for one hour.

This produced a dark brown metallic residue which gave a strong test for H_2S, and no test for sulphuric acid, showing complete dissociation of the sulphate.

Experiment 11: — A sample of the above mixture treated as in Experiment 9 to a temperature of 750°C. for one hour; complete dissociation resulting.

Experiment 12: — The same as Experiments 9 and 10, but raised to a temperature of 700°C. and held for one hour. No decomposition evident.

From these results it was evident: 1st, that clay, and a mixture of free sodium silicate and ferric iron, in the presence of a high percentage of CO. were effective in reducing the temperature of dissociation of gypsum: 2nd, that below 700°C. the rate of dissociation was very slow, both for a mixture of gypsum and clay and a mixture of gypsum, sodium silicate and ferric oxide: 3rd, that the dissociation point of pure gypsum is materially lowered in an atmosphere of CO, since 1200°C. is given as the dissociation temperature of $CaSO_4 2H_2O$ under atmospheric conditions: 4th, mixtures of 80 percent clay and 20 percent gypsum when heated to a temperature of 800°C., either in an atmosphere of CO alone, or in a mixture of CO and CO_2 in proportions given in the experiment, permit easy and rapid decomposition of the sulphate.

In practical operation, the calcium sulphate is more or less intimately mixed with clay, on the surface of clay pieces and exposed to fire gases existing during burning. The clay pieces must go thru an oxidizing fire at temperatures between 700°C. to 800°C. to prepare the clay for further burning. This preparation should be completed before any surface reduction is produced, as the latter would no doubt cause a more dense surface layer of clay at the temperature (750°-800°C.), and the rate of oxidation would be considerably decreased.

Bricks which were scummed in drying were burned in kilns in order to see if any action could be obtained. The first kiln was raised to 700°C. with oxidizing conditions, and the brick completely oxidized and then held between 700° and 800°C for six hours in a reducing atmosphere produced by a thick bed of hot coke and a very low draft. A gas analysis during this period showed, on the average, about 2 percent of O_2 and 1½ percent CO. This seemed to be the greatest reduction we could obtain. A second burn was made in which these conditions were repeated, except fuel oil was allowed to drop in the kiln during the reducing period. The gas analysis showed an increase of CO to 4 percent with the same amount of O_2.

After the reduction period, each kiln was burned to 1000°C. under oxidizing conditions and opened. There was apparently no improvement in the color.

Samples of clay pieces which were scummed during drying were placed in the combustion furnace and treated under the conditions of Experiment 5, then burned to 1000°C, under oxidizing conditions. Again, no improvement in the color was evident.

Other samples were cooled down immediately after treatment, and the scum examined. It seemed to have bonded with the clay to form a permanent discoloration.

Substitutes for Barium Carbonate. As there are a number of salts which react similarly to $BaCO_3$ in breaking up the calcium sulphate by forming an insoluble sulphate, a number of experiments were tried to determine the results which might be expected from their use in case they became commercially available. Also several other reagents were tried which might

TABLE I. THE FOLLOWING AMOUNTS OF REAGENTS WERE ADDED TO 300 GRAM SAMPLES OF CLAY MOULDED INTO BRICKETTES AND BURNED IN A CLEAN OXIDIZING FIRE:

REAGENT	AMOUNTS ADDED						RESULTS
	NO. 1	NO. 2	NO. 3	NO. 4	NO. 5	NO. 6	
2/10 Normal HCl.	5cc.	10cc.	15cc.	20cc.	25cc.	30cc.	No effect.
	percent	*percent*	*percent*	*percent*	*percent*	*percent*	
Starch	0.12	0.25	.5	.75	1	1.25	No effect.
Slaked lime.....	.24	.3	.37	.43	.47	.53	Scummed worse.
*Lignite solution..	5cc.	10cc.	15cc.	20cc.	25cc.	30cc.	Still scummed and showed darker color.
	percent	*percent*	*percent*	*percent*	*percent*	*percent*	
BaF_2	.1	.2	.25	.3	.35	.4	Nos. 3-4-5-6 free from scumming.
$BaCO_3$	.1	.2	.25	.3	.35	.4	Nos. 4-5-6 are free from scum.
$Ba(OH)_2$ $8H_2O$.. Ba. Hydrate.....	.1	.2	.25	.3	.35	.4	Nos. 4-5-6 free from scum. Red.
Sodium oxalate...	.1	.2	.3	.4	.5	.6	No. 3 improved; 4-5-6 no scum.
Oxalic acid.....	.1	.2	.25	.3	.35	.4	No results.
Sodium Silico-fluoride	.1	.2	.25	.3	.35	.4	No results.
Sodium fluoride....	.1	.2	.25	.3	.35	.4	Nos. 3-4-5-6 free from scumming.
Salt	.1	.2	.25	.3	.35	.4	Nos. 3, 4, 5 and 6 seemed free from scum but fluxed with clay.
Soap	.1	.2	.25	.3	.35	.4	No results. Produced pimpled surface and some sticking.

* Made by grinding 1 pound lignite in 2 gallons of water.

be expected to increase the adsorption of the clay and thus prevent the sulphate from traveling to the surface.

The accompanying table shows briefly the results obtained. The method of testing was to temper the clays with water containing the required amount of reagent, molding the clay into discs 3½ inches in diameter, by 1 inch thick and burning in an oil-fired kiln, free from reducing conditions. Samples containing barium carbonate were used in all cases for comparison.

The tests showed that BaF_2, $Ba(OH)_2\ 8H_2O$, $Na_2C_2O_4$, and NaF work equally as well as $BaCO_3$. The barium hydrate is being used commercially at present, and the use of barium fluoride has been previously mentioned by Professor Staley[1].

[1] The use of Barium Fluoride for the prevention of Dryer Scum on Bricks. Staley. Vol. 17, *Trans. Amer. Cer. Soc.*, page 200.

A METHOD OF TESTING THE CORROSIVE ACTION OF SLAGS ON FIRE BRICK[1]

BY G. H. BROWN

In power plant installations and many of the metallurgical processes, the slagging action of the ash, or other molten materials in contact with the refractory materials of the furnaces, gives rise to difficulties.

An indication of the type of refractory material which is best suited to withstand the action of a slag may, in some cases, be determined from chemical analysis of the refractory materials and of the slag. However, the slagging action is dependent to a certain extent on the physical structure of the refractory material, and selections based entirely on chemical analysis are apt to be misleading.

Character of Slagging Action. The action of slags on fire brick may be said to be of two distinct types. First, the pentration of the slag into the pores of the brick by capillary action; second, the dissolving of the brick or portions of the brick by the chemical action of the slag. This dissolving action is evidenced by a honeycomb structure at the surface of a brick in case either the bonding material or the grog are dissolved by the slag, or by a smooth cutting away of the surface of the brick when both the bonding material and grog particles are dissolved by the slag.

Obviously, the lower the porosity or the greater the density of the brick the greater will be its resistance to the penetration of the slag, the surface action being more pronounced.

Methods of Testing Slagging Action. Several methods have been used in determining the probable action of slags on refractory materials, some of the ones more generally used being: 1. The placing of the slag in a depression which is molded or drilled into the surface of the brick and subjecting it to the fur-

[1] By permission of the Director, Bureau of Standards.

nace temperature; 2, the placing of the slag in a refractory box which is cemented to the surface of the brick; 3, measurement of the depth to which a viscous colored solution penetrates into the brick.

According to our observations, there are some objections to the first method of placing the slag in a depression in the brick. The quantity of slag which may be used is small, and if there is chemical action of the slag on the refractory material the chemical composition is changed causing increases or decreases in the viscosity of the slag and giving rise to erroneous conclusions. Aside from this, the proportion of the surface area of the brick which is subjected to the slagging action is small and imperfections in the structure of the brick may be undetected. A further objection which is sometimes raised to the drilling of a cavity in the brick is the destruction of the outer skin of the brick thereby increasing the ease of slag penetration.

The objections to the method of placing the slag in a receptacle cemented to the surface of the brick are, for the most part, the same as those to method one, although in addition we have difficulties arising from the failure of the receptacle to adhere to the surface of the brick and the consequent loss of the slag.

The use of a colored solution to determine the action of the slag must be accompanied by a heating test of the brick in contact with the slag in order to determine the chemical action of the slag on the brick.

Types of Slag Investigated. The tests described were made on an ash from a coal which afforded difficulties in firing, on account of the violent action of the slag on the refractories used in the construction of the arches and bridge walls. The use of a refractory whose chemical composition would appear to offer the greatest resistance to the action of the slag did not improve conditions, and actual slagging tests were resorted to.

The chemical composition of the slag was as follows:

	Percent.
Silica	34.19
Alumina	14.63
Ferric oxide	32.14

Calcium oxide	7.55
Magnesium oxide	0.94
Potassium oxide	1.47
Sodium oxide	1.14
Sulphur trioxide	6.53

The softening temperature of the slag, as determined by the fuel laboratory of the Bureau of Mines, was found to be 1279-1305°C, under oxidizing conditions and 1086-1119°C, under reducing conditions.

Description of the Test. Fire-clay boxes whose inside dimensions were 9-in. length, 8-in. width and 3-in. depth, were molded from a finely ground refractory mixture and burned to cone 12. Each brick to be tested was placed on edge in the box and firmly packed with the finely ground slag on the two sides. In the first series of tests, slag prepared from the ash of the coal was used. In the later tests, the brick were subjected to the action of a synthetic mixture having a composition approaching that of the slag.

Twelve of the boxes were placed in a down-draft kiln fired with natural gas. The temperature was increased to 1400°C, over a period of 36 hours. For this slag, reducing conditions were maintained in the kiln over the last 12 hours of the burning. Twenty-four brands of brick of widely varying composition were tested under the corrosive action of the slag. In order to observe the corrosive action and penetration of the slag, each box and contained brick was broken into two or more parts.

In the illustration, Figure 1, is shown the action of the slag on several types of refractories. Taking into consideration the basic character of the slag, its action on the different types of refractories was somewhat unexpected.

The slag vigorously attacked a silica brick penetrating to the center and producing a honeycomb structure. The action on a bauxite brick was very similar to that on the silica brick, the slag penetrating to the center and rapidly dissolving the matrix of the brick. A magnesite brick shows penetration to the center and considerable dissolving action at the surfaces. The action on a carborundum brick is unlike that on any of the types of refrac-

tories tested. Although excessive surface-dissolving action has taken place but little or no penetration is noted. The porosity of the carborundum brick, twenty-one percent, was greater than that of any of the brick tested, and the action of the slag was not expected. The action of the slag on the different brands of clay brick varied somewhat with the composition and the structure of the brick. The so-called quartzite brick and brick containing a high percentage of silica were more or less vigorously attacked by the slag.

Among the clay brick tested were found several brands which appeared to resist both the dissolving action and the penetration of the slag. However, structural imperfections in some of the clay brick permitted irregular penetration of the slag, although the dissolving action appeared to be slight. This was observed in soft burned clay brick containing coarsely ground grog.

Conclusions. It would appear that this method of testing the corrosive action of a slag has the following advantages:

1. A considerable portion of the brick is subjected to the action of the slag. Structural imperfections of the brick are detected which may not be observed by testing only a small section of the brick.

2. The use of a large quantity of slag reduces the possibility of appreciable changes in the chemical and physical properties which may occur when smaller amounts of slag are used.

3. By the use of a large amount, the time effect of the slagging action on the brick may be studied. This is not possible when a small amount of slag is placed on the surface of a brick, especially in cases where the penetration and dissolving action is very rapid.

Obviously in installations employing high temperatures, tests as to slagging action must be accompanied by tests as to the refractoriness of the material. The ability of a type of refractory to withstand the action of slag may be due to its lack of refractoriness and consequent vitrified structure. A material of this kind, although able to resist the corrosive action of the slag at ordinary temperatures, may be too low in refractoriness to resist the furnace temperatures employed.

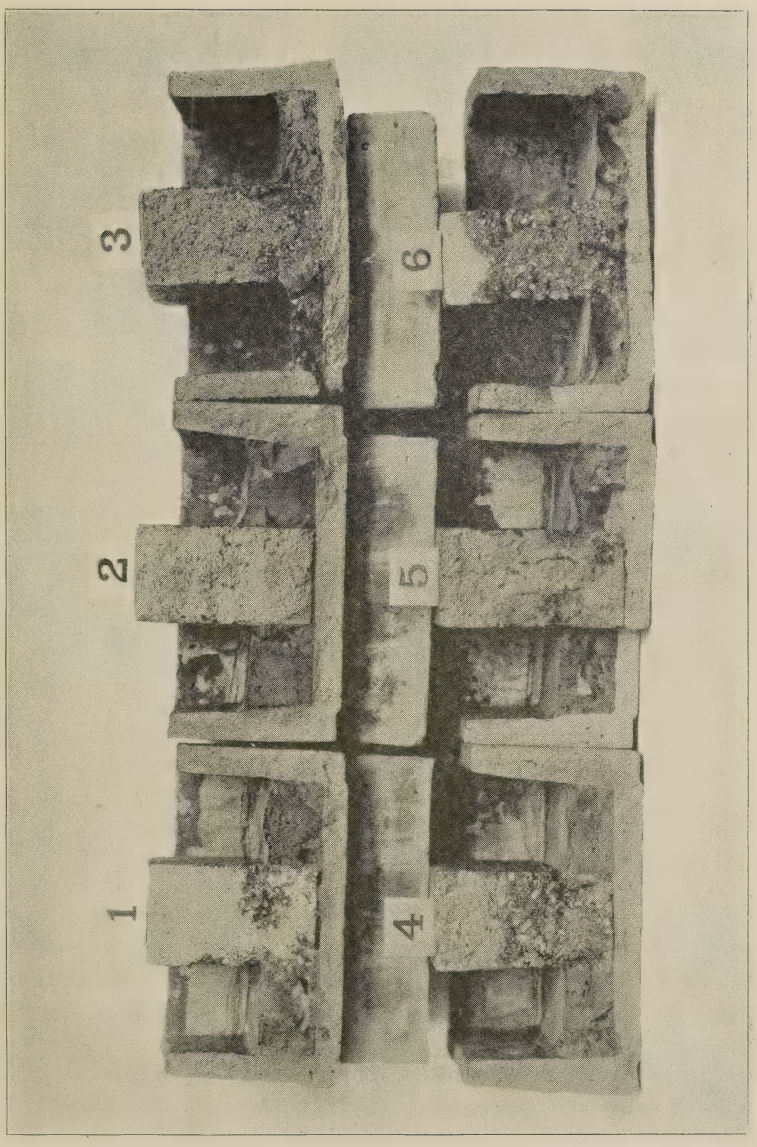


FIG. 1.

INFLUENCE OF VARIABLE SILICA AND ALUMINA ON TERRA COTTA GLAZES

BY R. B. KEELER

It is the opinion of the writer that we cannot safely assume any particular ratio between silica and alumina to be correct for all RO combinations. Furthermore, we cannot state definitely that any RO which we may select is useless simply because it does not make a good mat or bright glaze with a given ratio between silica and alumina.

Whatever RO we select will form a glaze if the proper proportions of silica and alumina are present and the glaze is given the proper heat treatment, so long as we do not deviate too widely in the selection of the RO.

For ordinary terra cotta glazes it is safe to follow the limits given below in selecting the RO.

	molecules
KNaO	.2 to .4
CaO	.1 to .6
ZnO	.1 to .4
MgO	.02 to .1
BaO	.02 to .3
PbO	.0 to .6
Al_2O_3	.2 to .5
SiO_2	1.5 to .4
SnO_2	.02 to .15

In the following groups we have selected a number of RO combinations according to the type of glaze required. The silica and alumina are varied to determine the best combination of the base, intermediary and acid which will produce satisfactory mats and glazes. The silica is low along the left side of the group and increased toward the right side. The alumina is low at the bottom and increased toward the top thus allowing the silica and alumina to cross in opposite directions.

Batches of each of the four corners of the groups were prepared and after being wet ground in pebble mills for two hours, the members were made up by cross-blending.

The glazes were sprayed over a white underslip on an ordinary No. 2 fire clay body containing about 40 percent grog.

Trials were fired in commercial 20 foot muffle kilns; burning about 50 to 70 hours. Temperature ranging between cones 3 and 5. Oil used as fuel.

Group R — Cone 2

Formula

$$\left.\begin{matrix} .15\ K_2O \\ .30\ CaO \\ .25\ ZnO \\ .10\ MgO \\ .10\ BaO \\ .10\ PbO \end{matrix}\right\} .2 \text{ to } .35\ Al_2O_3\ 1.5 \text{ to } 3.0\ SiO_2$$

High Al_2O_3 Low SiO_2				High Al_2O_3 High SiO_2
No. 1 .35 Al_2O_3 1.5 SiO_2	No. 2 .35 Al_2O_3 1.875 SiO_2	No. 3 .35 Al_2O_3 2.25 SiO_2	No. 4 .35 Al_2O_3 2.625 SiO_2	No. 5 .35 Al_2O_3 3.0 SiO_2
No. 6 .3125 1.5	No. 7 .3125 1.875	No. 8 .3125 2.25	No. 9 .3125 2.625	No. 10 .3125 3.
No. 11 .275 1.5	No. 12 .275 1.875	No. 13 .275 2.25	No. 14 .275 2.625	No. 15 .275 3.
No. 16 .2375 1.5	No. 17 .2375 1.875	No. 18 .2375 2.25	No. 19 .2375 2.625	No. 20 .2375 3.
No. 21 .2 1.5	No. 22 .2 1.875	No. 23 .2 2.25	No. 24 .2 2.625	No. 25 .2 3.
Low Al_2O_3 Low SiO_2				Low Al_2O_3 High SiO_2

Batch Weights

	No. 1	No. 5	No. 21	No. 25
Spar	83.55	83.55	83.55	83.55
Whiting	30.	30.	30.	30.
Zinc oxide	20.25	20.25	20.25	20.25
$MgCO_3$	8.4	8.4	8.4	8.4
$BaCO_3$	19.7	19.7	19.7	19.7
$PbCO_3$	25.8	25.8	25.8	25.8
Ball clay	51.6	51.6	12.9	12.9
Flint	12.	102.	30.	120.
	251.3	341.3	230.6	320.6

Results

No. 1 Fine dull mat	No. 2 Fine semi-mat	No. 3 Fine Semi-mat	No. 4 Little rough	No. 5 Immature
No. 6 Fine dull mat	No. 7 Fine semi mat	No. 8 Fine enamel	No. 9 Good glaze, a little rough	No. 10 Immature
No. 11 Fine dull mat Best	No. 12 Fine enamel	No. 13 Fine semi-mat	No. 14 Little rough	No. 15 Immature
No. 16 Fine mat smooth	No. 17 Fine enamel dull	No. 18 Fine enamel	No. 19 Little rough	No. 20 Immature
No. 21 Good dull enamel	No. 22 Good enamel quite dull	No. 23 Dull enamel	No. 24 Immature	No. 25 Immature

Color of members slightly cream. Texture ranging from dull mats to fair glazes but is inclined to be more of the semi-mat type.

Trials of this group were also sprayed on the body without underslip and all trials were equally as good in texture as those sprayed over the underslip. Color was slightly cloudy evidently with crystalline matter in suspension, giving glaze a fairly light color.

Samples flowed well, leaving no indication of waviness. Glaze over the underslip rounded out well at the edges and showed no thinness.

Norwegian feldspar, English ball clay and German flint were used.

Group S — Cone 3

Formula

$$\left.\begin{matrix} .2\ K_2O \\ .3\ CaO \\ .11\ MgO \\ .39\ ZnO \end{matrix}\right\} .35 \text{ to } 48\ Al_2O_3\ 1.5 \text{ to } 3.5\ SiO_2$$

High Al_2O_3 Low SiO_2				High Al_2O_3 High SiO_2
No. 1 .48 Al_2O_3 1.5 SiO_2	No. 2 .48 2.	No. 3 .48 2.5	No. 4 .48 3.	No. 5 .48 3.5
No. 6 .4475 1.5	No. 7 .4475 2.	No. 8 .4475 2.5	No. 9 .4475 3.	No. 10 .4475 3.5
No. 11 .415 1.5	No. 12 .415 2.	No. 13 .415 2.5	No. 14 .415 3.	No. 15 .415 3.5
No. 16 .3825 1.5	No. 17 .3825 2.	No. 18 .3825 2.5	No. 19 .3825 3.	No. 20 .3825 3.5
No. 21 .35 1.5	No. 22 .35 2.	No. 23 .35 2.5	No. 24 .35 3.	No. 25 .35 3.5
Low Al_2O_3 Low SiO_2				Low Al_2O_3 High SiO_2

Batch Weights

	No. 1	No. 5	No. 21	No. 25
Feldspar	668.4	445.6	668.4	445.6
Whiting	180.	120.	180.	120.
$MgCO_3$	55.4	36.96	55.44	36.96
Zinc oxide	189.54	126.36	189.54	126.36
Ball clay	232.2	288.96	232.2	154.8
$Al(OH)_3$	60.84	.....	.....	.....
Flint	.....	417.6	.....	480.
	1386.	1435.	1325.	1363.

Results

No. 1 Immature.	No. 2 Immature.	No. 3 Immature.	No. 4 Little glossy.	No. 5 Immature
No. 6 Immature.	No. 7 Runs a little. Mat.	No. 8 Runs a little. Glossy.	No. 9 Glossy. Runs.	No. 10 A little glossy.
No. 11 Immature.	No. 12 Runs a little. Mat.	No. 13 Runs a little. Glossy.	No. 14 Glossy. Runs.	No. 15 A little glossy.
No. 16 Immature mat.	No. 17 Immature. Mat.	No. 18 Runs together very well.	No. 19 Part matured.	No. 20 A little glossy.
No. 21 Immature mat.	No. 22 Semi-mat. Best of group.	No. 23 Next best of group— Bright.	No. 24 Fair— not run together.	No. 25 A little glossy.

Nearly all glazes in this group were immature, but had a good texture wherever partly matured. The bottom of the group looks very good and is almost fused over giving a partial mat texture.

It is quite possible that going lower with another field the fusibility would increase, but noting the high content of MgO (*i. e.* .11 molecules) it was decided not to continue lower. Possibly two cones higher in temperature would have fused them.

The low content of spar was noted, and it was decided to try higher spar content and omit the MgO altogether. We would probably sacrifice whiteness by increasing the fusibility.

Group T — Cone 4

Formula

$$\left.\begin{matrix} .4\ K_2O \\ .324\ ZnO \\ .275\ CaO \end{matrix}\right\} .4 \text{ to } .5\ Al_2O_3\ 2.4 \text{ to } 3.4\ SiO_2$$

High Al_2O_3 Low SiO_2				High Al_2O_3 High SiO_2
No. 1 .5 Al_2O_3 2.4 SiO_2	No. 2 .5 2.65	No. 3 .5 2.9	No. 4 .5 3.15	No. 5 .5 3.4
No. 6 .475 2.4	No. 7 .475 2.65	No. 8 .475 2.9	No. 9 .475 3.15	No. 10 .475 3.4
No. 11 .45 2.4	No. 12 .45 2.65	No. 13 .45 2.9	No. 14 .45 3.15	No. 15 .45 3.4
No. 16 .425 2.4	No. 17 .425 2.65	No. 18 .425 2.9	No. 19 .425 3.15	No. 20 .425 3.4
No. 21 .4 2.4	No. 22 .4 2.65	No. 23 .4 2.9	No. 24 .4 3.15	No. 25 .4 3.4
Low Al_2O_3 Low SiO_2				Low Al_2O_3 High SiO_2

Batch Weights

	No. 1	No. 5	No. 21	No. 25
Spar	1114.	891.	1114.	891.
Whiting	137.	110.	137.	110.
Zinc oxide	131.	104.	131.	104.
$Al(OH)_3$	39.	....	....	....
Flint	....	192.	....	240.
Ball clay	....	103.	....	....
	1421.	1401.	1382.	1346.

Results

No. 1 Good. Nearly mat.	No. 2 Good. Smooth.	No. 3 Good. Smooth.	No. 4 Little wavy.	No. 5 Beads.
No. 6 Good. Nearly mat.	No. 7 Good, nearly smooth.	No. 8 Good. Smooth.	No. 9 Little wavy.	No. 10 Little wavy. Clearer.
No. 11 Good semi-mat.	No. 12 Good semi-mat.	No. 13 Good, nearly clear.	No. 14 Good, nearly clear. Little wavy.	No. 15 Good, nearly clear. Crazed.
No. 16 Good semi-mat.	No. 17 Good semi-mat.	No. 18 Good, nearly clear.	No. 19 Good, nearly clear. Wavy.	No. 20 Too wavy, crazed.
No. 21 Good semi-mat.	No. 22 Good semi-mat.	No. 23 Fine, nearly clear.	No. 24 Fine, nearly clear.	No. 25 Nearly clear. Crazed.

Samples were nearly all good; fusing into clear bright glazes along the center of the field and were partially mat along left side of field. Color a creamy white.

Glazes on left side are by far the best, being almost white mats, while on the right-hand side they become clearer and finally craze, especially toward the low alumina and high silica end. All these glazes have an inclination to bead and craze where applied without underslip. Brandywine spar, German flint and English ball clay were used for this group.

Group T shows that high content of spar fuses glazes more readily but does not give a perfectly dull mat. Later on groups were made to the left of this series to determine whether mats could be produced with the given RO (See BO, CR and CA groups).

Group X — Cone 4

Formula

$$\left.\begin{matrix} .2\ K_2O \\ .15\ MgO \\ .30\ ZnO \\ .35\ CaO \end{matrix}\right\} .4 \text{ to } .48\ Al_2O_3\ 1.75 \text{ to } 3\ SiO_2$$

High Al_2O_3 Low SiO_2				High Al_2O_3 High SiO_2
No. 1 .48 Al_2O_3 1.75 SiO_2	No. 2 .48 2.06	No. 3 .48 2.375	No. 4 .48 2.687	No. 5 .48 3.
No. 6 .46 1.75	No. 7 .46 2.06	No. 8 .46 2.375	No. 9 .46 2.687	No. 10 .46 3.
No. 11 .44 1.75	No. 12 .44 2.06	No. 13 .44 2.375	No. 14 .44 2.687	No. 15 .44 3.
No. 16 .42 1.75	No. 17 .42 2.06	No. 18 .42 2.375	No. 19 .42 2.687	No. 20 .42 3.
No. 21 .4 1.75	No. 22 .4 2.06	No. 23 .4 2.375	No. 24 .4 2.687	No. 25 .4 3.
Low Al_2O_3 Low SiO_2				Low Al_2O_3 High SiO_2

Batch Weights

	No. 1	No. 5	No. 21	No. 25
Spar	557.	445.	668.	445.
Whiting	175.	140.	210.	140.
Zinc oxide	121.	97.	145.	97.
$MgCO_3$	63.	50.	75.	50.
Ball clay	129.	103.	154.	103.
China clay	232.	185.	154.	103.
Flint	....	297.	54.	336.
	1277.	1319.	1463.	1275.

Results

No. 1 Fine mat, Crazed.	No. 2 Fine mat, crazed.	No. 3 Mat, little rough.	No 4 Medium mat.	No. 5 Too wavy.
No. 6 Fine mat, crazed.	No. 7 Fine mat, crazed.	No. 8 Good mat.	No. 9 Good mat, little wavy.	No. 10 Nearly glossy, too wavy.
No. 11 Mat, crazed.	No. 12 Fine mat.	No. 13 Fine mat texture, white.	No. 14 Fine mat, a little wavy.	No 15 Semi-mat, wavy.
No. 16 Mat, crazed.	No. 17 Fine mat.	No. 18 Fine mat, little glossy.	No. 19 Fine semi-mat.	No. 20 Little wavy.
No. 21 Mat, wavy, crazed.	No. 22 Mat, little rough.	No. 23 Fine semi-mat.	No. 24 Little wavy.	No. 25 Little wavy.

This group was tried to secure a harder and whiter glaze than any of the T group. Whiteness is very good. Number 18 being about the center of the good mats. Perhaps if the MgO content had been cut down to .1 equivalent, the glaze would have been a little softer and would have produced a few bright glazes.

Although ZnO or MgO used singly in large quantities (over .4 equivalent) in a glaze will produce beading, it was noted in adding both MgO and ZnO that as much as .45 molecules made up of the two could be added without signs of beading.

In the next groups, it was decided to add a small amount of BaO to lower the fusion temperature and keep the K_2O as high as possible. Also to make the sum total of ZnO and MgO equal .4 in order to impart sufficient white color to the glaze. To increase whiteness still more, it was decided to add .02 SnO_2.

Group AE — Cone 4

Formula

$$\left.\begin{matrix} .38\ K_2O \\ .35\ ZnO \\ .17\ CaO \\ .05\ MgO \\ .05\ BaO \end{matrix}\right\} .4 \text{ to } .5\ Al_2O_3\ 2.4 \text{ to } 3.4\ SiO_2\ .02\ SnO_2$$

High Al_2O_3 Low SiO_2				High Al_2O_3 High SiO_2
No. 1 .5 Al_2O_3 2.4 SiO_2	No. 2 .5 2.65	No. 3 .5 2.9	No. 4 .5 3.5	No. 5 .5 3.4
No. 6 .475 2.4	No. 7 .475 2.65	No. 8 .475 2.9	No. 9 .475 3.15	No. 10 .475 3.4
No. 11 .45 2.4	No. 12 .45 2.65	No. 13 .45 2.9	No. 14 .45 3.15	No. 15 .45 3.4
No. 16 .425 2.4	No. 17 .425 2.65	No. 18 .425 2.9	No. 19 .425 3.15	No. 20 .425 3.4
No. 21 .4 2.4	No. 22 .4 2.65	No. 23 .4 2.9	No. 24 .4 3.15	No. 25 .4 3.4
Low Al_2O_3 Low SiO_2				Low Al_2O_3 High SiO_2

Batch Weights

	No. 1	No. 5	No. 21	No. 25
Spar	1058.	1058.	1058.	1058.
Whiting	85.	85.	85.	85.
Zinc oxide	141.	141.	141.	141.
$MgCO_3$	21.	21.	21.	21.
$BaCO_3$	49.	49.	49.	49.
Ball clay	77.	154.	25.	25.
$Al(OH)_3$	23.	....	....	....
SiO_2	....	264.	24.	324.
SnO_2	14.	14.	14.	14.
	1470.	1788.	1419.	1719.

Results

No. 1 Fine mat.	No. 2 Fine semi-mat.	No. 3 Fine semi-mat.	No. 4 Brighter, little wavy.	No. 5 Fair wavy.
No. 6 Fine mat.	No. 7 Fine semi-mat.	No. 8 Fine semi-mat, brighter.	No. 9 Bright, a little wavy.	No. 10 Bright and wavy.
No. 11 Fine mat.	No. 12 Fine semi-mat.	No. 13 Fine semi-mat. Little wavy.	No. 14 Fine semi-mat. Wavy.	No. 15 Too wavy.
No. 16 Fine mat.	No. 17 Fine, nearly clear.	No. 18 Clearer but wavy.	No. 19 Wavy.	No. 20 Too wavy.
No. 21 Fine, nearly clear.	No. 22 Fine, nearly clear.	No. 23 Clear but little wavy.	No. 24 Clear but still wavy.	No. 25 Too wavy.

Samples Nos. 6, 11 and 16 were most perfect, no crazing being evident in any of the glazes in this group. Norwegian spar,

English ball clay and German flint were used. If Brandywine spar had been used the result no doubt would have been quite different on account of the fusibility of Brandywine over Norwegian spar.

It was now decided to increase the number of components in the RO and the following groups were made.

Group AH — Cone 4

Formula

$$\left.\begin{matrix} .15\ K_2O \\ .20\ CaO \\ .25\ ZnO \\ .10\ MgO \\ .10\ BaO \\ .20\ PbO \end{matrix}\right\} .2 \text{ to } .35\ Al_2O_3\ 1.5 \text{ to } 3\ SiO_2$$

High Al_2O_3 Low SiO_2				High Al_2O_3 High SiO_2
No. 1 .35 Al_2O_3 1.5 SiO_2	No. 2 .35 1.875	No. 3 .35 2.25	No. 4 .35 2.625	No. 5 .35 3.
No. 6 .3125 1.5	No. 7 .3125 1.875	No. 8 .3125 2.25	No. 9 .3125 2.625	No. 10 .3125 3.
No. 11 .275 1.5	No. 12 .275 1.875	No. 13 .275 2.25	No. 14 .275 2.625	No. 15 .275 3.
No. 16 .2375 1.5	No. 17 .2375 1.875	No. 18 .2375 2.25	No. 19 .2375 2.625	No. 20 .2375 3.
No. 21 .2 1.5	No. 22 .2 1.875	No. 23 .2 2.25	No. 24 .2 2.625	No. 25 .2 3.
Low Al_2O_3 Low SiO_2				Low Al_2O_3 High SiO_2

Batch Weights

	No. 1	No. 5	No. 21	No. 25
Spar	417.	417.	417.	417.
Whiting	100.	100.	100.	100.
Zinc oxide	101.	101.	101.	101.
$MgCO_3$	42.	42.	42.	42.
$BaCO_3$	98.	98.	98.	98.
$PbCO_3$	258.	258.	258.	258.
Ball clay	129.	129.	64.	64.
China clay	129.	129.	....	....
Flint	60.	510.	150.	585.
Tin oxide	22.	22.	22.	22.

Results

No. 1 Good mat.	No. 2 Fine mat.	No. 3 Good glaze, clear.	No. 4 Good, clear glaze.	No. 5 Very rough.
No. 6 Fine mat.	No. 7 Fine semi-mat.	No. 8 Fair, clear.	No. 9 Clear, wavy.	No. 10 Very rough.
No. 11 Mat Grueby Texture.	No. 12 Clear, too wavy.	No. 13 Clear, too wavy.	No. 14 Clear, too wavy.	No. 15 Dull, Immature.
No. 16 Grueby semi-mat.	No. 17 Too rough.	No. 18 Too rough.	No. 19 Too rough.	No. 20 Dull, Immature.
No. 21 Too rough.	No. 22 Too rough.	No. 23 Too rough.	No. 24 Too rough.	No. 25 Too rough.

The glazes were cream in color with a few good mats. Clear glazes were a little wavy, which may possibly be due to the low spar content.

Trials along the high silica side of the group are profusely cracked and are very immature.

By following this group up to the left corner with another, increasing the alumina up to .5 molecules would probably bring some good results.

It was now decided to go to the left of the T group to ascertain the limits of this field. No good mats were evident along the left side of the T group, and it was believed that by going further to the left we would find mats.

Group BO — Cone 4

In this group it is impossible to keep the RO constant.

Formula

RO .3 to .4 Al_2O_3 2.4 to 3.4 SiO_2

	No. 1	No. 2	No. 3	No. 4	No. 5
.35 K_2O .25 CaO .30 ZnO .05 MgO .05 BaO	.4 2.4	.4 2.65	.4 2.9	.4 3.15	.4 3.4
	No. 6	No. 7	No. 8	No. 9	No. 10
.30 K_2O .30 CaO .30 ZnO .05 BaO .05 MgO	.35 2.4	.35 2.65	.35 2.9	.35 3.15	.35 3.4
	No. 11	No. 12	No. 13	No. 14	No. 15
.25 K_2O .35 CaO .30 ZnO .05 MgO .05 BaO	.3 2.4	.3 2.65	.3 2.9	.3 3.15	.3 3.4

Batch Weights

	No. 1	No. 5	No. 11	No. 15
Spar	584.	584.	417.	417.
Whiting	75.	75.	105.	105.
Zinc oxide	72.	72.	72.	72.
$MgCO_3$	12.	12.	12.	12.
$BaCO_3$	29.	29.	29.	29.
Ball clay	38.	38.	38.	38.
Flint	36.	72.	144.	324.

Results

No. 1 Fine clear glaze, slight matness.	No. 2 Clear glaze, slight matness.	No. 3 Clear glaze, slight matness.	No. 4 Clear glaze, slight matness.	No. 5 Clear glaze, slight matness.
No. 6 Clear.	No. 7 Clear.	No. 8 Clear.	No. 9 Clear, little wavy.	No. 10 Clear, wavy.
No. 11 Fine, clear, slightly wavy.	No. 12 Fine, clear, slightly wavy.	No. 13 Wavy.	No. 14 Not mature.	No. 15 Quite rough.

Results were a disappointment as far as mats were concerned, but glazes were perfect, especially BO No. 6. Another glaze was tried still further to the left of the BO group.

BP Glaze — Cone 4

In the following glazes Al_2O_3 was lowered for matness.

Formula

$$\left.\begin{matrix} .30\ K_2O \\ .36\ ZnO \\ .34\ CaO \end{matrix}\right\} .4\ Al_2O_3\ 2.0\ SiO_2$$

Batch Weights

Spar	167
Whiting	34
Zinc oxide	29
Ball clay	26
	256

Results

Glaze was a very good mat except too rough, rather pinholed and lighter in color than most of the glazes of its kind.

Glaze BZ — Cone 4

Formula

$$\left.\begin{matrix}.3\ K_2O \\ .3\ ZnO \\ .06\ MgO \\ .03\ BaO \\ .31\ CaO\end{matrix}\right\} .4\ Al_2O_3\ 2.\ SiO_2\ .03\ SnO_2$$

Batch Weights

Spar	167
Whiting	31
Zinc oxide	29
$MgCO_3$	5
$BaCO_3$	6
Ball clay	13
China clay	13
	259

Glaze BZ was made up using a different RO, replacing part of the ZnO with MgO and CaO with a very small amount of BaO.

Results. Glaze a fine, smooth mat, good white and runs a little. CA was then tried, which was a continuation to the left of the T group, using higher alumina than in the BO group.

Group CA — Cone 4

.13 K_2O .46 CaO .33 ZnO .05 MgO .03 BaO	No. 1 .5 Al_2O_3 1.5 SiO_2	No. 2 .5 1.8	No. 3 .5 2.1	K_2O .27 CaO .32 ZnO .33 MgO .05 BaO .03
	No. 4 .475 1.5	No. 5 .475 1.8	No. 6 .475 2.1	
	No. 7 .45 1.5	No. 8 .45 1.8	No. 9 .45 2.1	
	No. 10 .425 1.5	No. 11 .425 1.8	No. 12 .425 2.1	
.17 K_2O .42 CaO .33 ZnO .05 MgO .03 BaO	No. 13 .4 1.5	No. 14 .4 1.8	No. 15 .4 2.1	K_2O .32 CaO .27 ZnO .33 MgO .05 BaO .03

Batch Weights

	No. 1	No. 3	No. 13	No. 15
Spar	72.	150.	94.	178.
Whiting	46.	32.	42.	27.
Zinc oxide	27.	27.	27.	27.
$MgCO_3$	4.	4.	4.	4.
$BaCO_3$	6.	6.	6.	6.
Ball clay	48.	29.	29.	10.
China clay	47.	29.	29.	11.
Flint	....	1.	1.	1.
SnO_2	6.	6.	6.	6.

Results

No. 1 Immature.	No. 2 Immature.	No. 3 Fair, white mat, little rough.
No. 4 Immature.	No. 5 Rough mat.	No. 6 Good mat, white and smooth.
No. 7 Immature.	No. 8 Fair, white mat, little rough.	No. 9 Fair, semi-mat, little rough.
No. 10 Immature.	No. 11 Fair, white mat, little rough.	No. 12 Fine semi-mat, smooth.
No. 13 Immature.	No. 14 Fine white mat.	No. 15 Fine semi-mat, smooth.

It was impossible to keep the RO constant, in the above group, on account of the small amount of silica.

The mats in this group are fairly good but all show a slight roughness. CA — No. 12 is the whitest of this group.

Group CF — Cone 4

No. 1 .04 K_2O .54 CaO .32 ZnO .05 MgO .05 BaO .4 Al_2O_3 1. SiO_2	No. 2 .115 K_2O .465 CaO .32 ZnO .05 MgO .05 BaO .4 Al_2O_3 1.3 SiO_2	No. 3 .190 K_2O .39 CaO .32 ZnO .05 MgO .05 BaO .4 Al_2O_3 1.6 SiO_2	No. 4 .265 K_2O .315 CaO .32 ZnO .05 MgO .05 BaO .4 Al_2O_3 1.9 SiO_2	No. 5 .34 K_2O .24 CaO .32 ZnO .05 MgO .05 BaO .4 Al_2O_3 2.2 SiO_2
No. 6 .07 K_2O .51 CaO .32 ZnO .05 MgO .05 BaO .35 Al_2O_3 1. SiO_2	No. 7 .12625 .45375 .32 .05 .05 .35 Al_2O_3 1.3 SiO_2	No. 8 .1825 .3975 .32 .05 .05 .35 Al_2O_3 1.6 SiO_2	No. 9 .23875 .34125 .32 .05 .05 .35 Al_2O_3 1.9 SiO_2	No. 10 .295 .285 **.32** .05 .05 **.35** Al_2O_3 2. 2 SiO_2
No. 11 .10 K_2O .48 CaO .32 ZnO .05 MgO .05 BaO .3 Al_2O_3 1. SiO_2	No. 12 .1375 .4425 .32 .05 .05 .3 Al_2O_3 1.3 SiO_2	No. 13 .175 .405 .32 .05 .05 .3 Al_2O_3 1.6 SiO_2	No. 14 .2125 .3675 .32 .05 .05 .3 Al_2O_3 1.9 SiO_2	No. 15 .25 .33 **.32** .05 .05 .3 Al_2O_3 2.2 SiO_2

Batch Weights

	No. 1	No. 5	No. 11	No. 15
Spar	22.28	189.38	55.7	139.25
Whiting	54.	24.	48.	33.
Zinc oxide	25.92	25.92	25.92	25.92
$MgCO_3$	4.2	4.2	4.2	4.2
$BaCO_3$	9.8	9.8	9.8	9.8
Ball clay	15.48	15.48	51.6	12.9
Flint	38.4	2.4	....	36.

Results

No. 1 Immature.	No. 2 Little lumpy.	No. 3 Good, white fairly smooth.	No. 4 Good semi-mat, cream color.	No. 5 Good semi-mat, cream color.
No. 6 Immature.	No. 7 Grueby texture.	No. 8 Semi-mat, fair texture, cream color.	No. 9 Semi-mat, fair texture.	No. 10 Good semi-mat, cream color.
No. 11 Very immature.	No. 12 Rough.	No. 13 Good cream color, semi-mat.	No. 14 Nearly clear, little wavy.	No. 15 Clear glaze, little wavy, crazed.

This group is a continuation of the BO group; going to the left for mats and replacing K_2O with CaO. This group did not mat up as much as expected, although there are some very good semi-mats. There is a possibility of going lower for clear glazes between this group and the BO group. In the above group it was impossible to keep the RO constant on account of low silica content.

Group CR — Cone 4

Impossible to keep the RO constant on account of low Al_2O_3.

.21 K_2O .37 CaO .32 ZnO .05 MgO .05 BaO						Constant .12 SnO_2
	No. 1 .28 2.	No. 2 .28 2.3	No. 3 .28 2.6	No. 4 .28 2.9	No. 5 .28 3.2	
	No. 6 .24 2.	No. 7 .24 2.3	No. 8 .24 2.6	No. 9 .24 2.9	No. 10 .24 3.2	
.16 K_2O .42 CaO .32 ZnO .05 MgO .05 BaO						
	No. 11 .2 2.	No. 12 .2 2.3	No. 13 .2 2.6	No. 14 .2 2.9	No. 15 .2 3.2	

Batch Weights

	No. 1	No. 5	No. 11	No. 15
Spar	117.	117.	89.	89.
Whiting	37.	37.	42.	42.
Zinc oxide	26.	26.	26.	26.
$MgCO_3$	4.	4.	4.	4.
$BaCO_3$	10.	10.	10.	10.
Ball clay	18.	18.	10.	10.
Flint	36.	108.	57.	129.
SnO_2	18.	18.	18.	18.

Results

No. 1 Cream, semi-mat.	No. 2 Cream, semi-mat.	No. 3 Little immature.	No. 4 Immature.	No. 5 Immature.
No. 6 Cream, semi-mat, wavy.	No. 7 Immature.	No. 8 Immature.	No. 9 Immature.	No. 10 Immature.
No. 11 Immature.	No. 12 Immature.	No. 13 Immature.	No. 14 Immature.	No. 15 Immature.

Continuing lower than the BO group, it was noted that it is almost impossible to obtain satisfactory results by adding SnO_2 to a good alkali glaze without altering the glaze decidedly. The glazes invariably become wavy and do not flow readily. They present every appearance of being too high in alumina. It is evident in this type of glaze that the Al_2O_3 should be lowered when SnO_2 is introduced.

Group DK — Cone 4

Formula

$$\left.\begin{matrix} .25\ K_2O \\ .36\ ZnO \\ .06\ MgO \\ .33\ CaO \end{matrix}\right\} .3 \text{ to } .5\ Al_2O_3 \Big\} 2. \text{ to } 3.2\ SiO_2 \quad .05\ SnO_2$$

High Al_2O_3 Low SiO_2				High Al_2O_3 High SiO_2
No. 1 .5 Al_2O_3 2. SiO_2	No. 2 .5 2.3	No. 3 .5 2.6	No. 4 .5 2.9	No. 5 .5 3.2
No. 6 .45 2.	No. 7 .45 2.3	No. 8 .45 2.6	No. 9 .45 2.9	No. 10 .45 3.2
No. 11 .4 2.	No. 12 .4 2.3	No. 13 .4 2.6	No. 14 .4 2.9	No. 15 .4 3.2
No. 16 .35 2.	No. 17 .35 2.3	No. 18 .35 2.6	No. 19 .35 2.9	No. 20 .35 3.2
No. 21 .3 2.	No. 22 .3 2.3	No. 23 .3 2.6	No. 24 .3 2.9	No. 25 .3 3.2
Low Al_2O_3 High SiO_2				Low Al_2O_3 High SiO_2

Batch Weights

	No. 1	No. 5	No. 21	No. 25
Spar	139	139	139	139
Whiting	33	33	33	33
Zinc oxide	29	29	29	29
$MgCO_3$	5	5	5	5
Ball clay	10	10	13	13
China clay	54	54	..	..
Flint	..	72	24	96
SnO_2	7	7	7	7

Results

No. 1 Immature.	No. 2 Immature.	No. 3 Immature.	No. 4 Immature.	No. 5 Fair mat, little rough.
No. 6 Immature.	No. 7 Immature.	No. 8 Good mat.	No. 9 Hard mat.	No. 10 Semi-mat, too hard.
No. 11 Mat, crazed.	No. 12 Mat, crazed.	No. 13 Good mat.	No. 14 Fine semi-mat.	No. 15 Fine, semi-mat.
No. 16 Mat, crazed.	No. 17 Mat, runs.	No. 18 Semi-mat, runs.	No. 19 Clear but rough.	No. 20 Clear but rough.
No. 21 Good, clear.	No. 22 Good, clear glaze.	No. 23 Fine, clear glaze.	No. 24 Good, clear glaze.	No. 25 Good clear glaze.

Group DK — Cone 4

In this group as much Cornish stone as possible was used. The same proportion of silica and alumina was used as in previous DK group.

This group has some fine white mats about the center and fine clear ones along the lower right hand corner. Color a very good white. Brandywine spar used.

RESULTS — GROUP DK — CONE 4

No. 1 Rough and finely beaded.	No. 2 Rough and finely beaded.	No. 3 Rough and finely beaded.	No. 4 Not as rough as No. 3.	No. 5 Little too rough for a mat.
No. 6 Rough but in larger lumps.	No. 7 Near to a mat but crazed.	No. 8 Good mat but wavy and crazed.	No. 9 Good mat, trifle wavy and white.	No. 10 Good mat, little more glossy than No. 9.
No. 11 Smoother than No. 6, crazed.	No. 12 Good mat, but crazed.	No. 13 Good white mat.	No. 14 Good white mat, little glossy.	No. 15 More glossy than No. 14, cream color.
No. 16 Smooth mat, crazed.	No. 17 Good white mat.	No. 18 Good strong mat, little glossy.	No. 19 Good semi-mat.	No. 20 Too rough—cream color.
No. 21 Good mat, but slightly pitted.	No. 22 Fine clear glaze, but crazed.	No. 23 Fine white enamel.	No. 24 Fine white enamel.	No. 25 Rough white mat, particles in suspension.

Group DK was made up using all Cornish stone possible. The best of this group for matness is No. 13 though undesirable as to smoothness, the surface being pitted. It is not as good as the spar mat. Number 24 is the best white enamel and shows a fine white color.

The use of Cornish stone in this group shows that it improves an enamel decidedly (at this temperature), but glazes are not so smooth as with spar. Cornish stone seems to impart a lower viscosity to a white glaze and causes it to flow over imperfections on the clay with much more ease than spar.

Group DM — Cone 4

Formula

$$\left.\begin{matrix} .2\ K_2O \\ .5\ CaO \\ .3\ ZnO \end{matrix}\right\} .25 \text{ to } .45\ Al_2O_3 \left\{ 2. \text{ to } 3.5\ SiO_2 \right. .05\ SnO_2$$

High Al_2O_3 Low SiO_2				High Al_2O_3 High SiO_2
No. 1 .45 Al_2O_3 2. SiO_2	No. 2 .45 2.375	No. 3 .45 2.75	No. 4 .45 3.125	No. 5 .45 3.5
No. 6 .4 2.	No. 7 .4 2.375	No. 8 .4 2.75	No. 9 .4 3.125	No. 10 .4 3.5
No. 11 .35 2.	No. 12 .35 2.375	No. 13 .35 2.75	No. 14 .35 3.125	No. 15 .35 3.
No. 16 .3 2.	No. 17 .3 2.375	No. 18 .3 2.75	No. 19 .3 3.125	No. 20 .3 3.5
No. 21 .25 2.	No. 22 .25 2.375	No. 23 .25 2.75	No. 24 .25 3.125	No. 25 .25 3.5
Low Al_2O_3 Low SiO_2				Low Al_2O_3 High SiO_2

Batch Weights

	No. 1	No. 5	No. 21	No. 25
Spar	111	111	111	111
Whiting	50	50	50	50
Zinc oxide	24	24	24	24
Ball clay	10	10	10	10
China clay	54	54	3	3
Flint	18	108	42	132
SnO_2	7	7	7	7

Results

RESULTS — GROUP DM — CONE 4

No. 1 Immature.	No. 2 Immature.	No. 3 Good mat but rough.	No. 4 Good mat but rough.	No. 5 Good mat but rough.
No. 6 Good mat but crazed.	No. 7 Alligator surface, good mat.	No. 8 Allgator surface, good mat.	No. 9 Fine semi-mat.	No. 10 Semi-mat. A little hard.
No. 11 Mat, crazed and pinholed.	No. 12 Fair mat, crazed.	No. 13 Fine semi-mat.	No. 14 Good semi-mat.	No. 15 Fair glaze, little rough.
No. 16 Mat, crazed and pinholed.	No. 17 Good clear mat.	No. 18 Good clear mat.	No. 19 Good clear mat.	No. 20 Rough and wavy.
No. 21 Too full of undissolved matter.	No. 22 Fair, clear.	No. 23 Fine semi-mat.	No. 24 Wavy.	No. 25 Rough.

This group has no pronounced mats but some very good semi-mats. The clear glazes are very good but have not the clear tone that glazes would have with more elements in the RO or less CaO. As for whiteness this group was very good.

Group DN — Cone 4

Formula

$$\left.\begin{matrix} .23\ K_2O \\ .40\ ZnO \\ .03\ MgO \\ .05\ BaO \\ .29\ CaO \end{matrix}\right\} .25 \text{ to } .45\ Al_2O_3 \left\{ 2. \text{ to } 3.5\ SiO_2 \right. \quad .05\ SnO_2$$

High Al_2O_3 Low SiO_2				High Al_2O_3 High SiO_2
No. 1 .45 Al_2O_3 2. SiO_2	No. 2 .45 2.375	No. 3 .45 2.75	No. 4 .45 3.125	No. 5 .45 3.5
No. 6 .4 2.	No. 7 .4 2.375	No. 8 .4 2.75	No. 9 .4 3.125	No. 10 .4 3.5
No. 11 .35 2.0	No. 12 .35 2.375	No. 13 .35 2.75	No. 14 .35 3.125	No. 15 .35 3.5
No. 16 .3 2.0	No. 17 .3 2.375	No. 18 .3 2.75	No. 19 .3 3.125	No. 20 .3 3.5
No. 21 .25 2.0	No. 22 .25 2.375	No. 23 .25 2.75	No. 24 .25 3.125	No. 25 .25 3.5
Low Al_2O_3 Low SiO_2				Low Al_2O_3 High SiO_2

Batch Weights

	No. 1	No. 5	No. 21	No. 25
Spar	128	128	128	128
Whiting	29	29	29	29
Zinc oxide	32	32	32	32
$MgCO_3$	2½	2½	2½	2½
$BaCO_3$	10	10	10	10
Ball clay	10	10	5	5
China clay	47	47	..	..
Flint	11	101	35	125
SnO_2	7	7	7	7

Results

No. 1 Hard mat, crazed.	No. 2 Good mat, hard.	No. 3 Alligator mat.	No. 4 Wavy, semi-mat.	No. 5 Wavy, nearly clear.
No. 6 Aligator mat, crazed.	No. 7 Mat, crazed.	No. 8 Rough.	No. 9 Fair — clear.	No. 10 Fair— clear.
No. 11 Slight segregation of particles.	No. 12 Crazed.	No. 13 Clear.	No. 14 Clear.	No. 15 Rough.
No. 16 Slight segregation of particles.	No. 17 Clear but pinholed.	No. 18 Clear.	No. 19 Clear.	No. 20 Fair— clear.
No. 21 Slight segregation of particles.	No. 22 Clear but pinholed.	No. 23 Clear.	No. 24 Clear.	No. 25 Good, clear.

This group shows no good mats but some very good semi-mats. The clear glazes lose some of their color due to the use of $BaCO_3$. Number 24 is best and a fine white. So far, the use of $BaCO_3$ for mats has not proved a success even with small

additions. Probably with a high content of $BaCO_3$ one could obtain a mat with suitable texture, but BaO added in small quantities tends to lower the fusibility especially in the presence of a large number of components in the RO. The more elements in the RO, the more it will dissolve SnO_2. Barium in the above series has a strong tendency to decrease white color.

In the usual experimental work it is quite evident that we should vary the base, intermediary and acid to obtain the desired results, and when we alter the base we should expect to alter the intermediary and acid.

We note in reviewing the groups that high alumina and low silica produce immature glazes in most cases, and where they do fuse they have a tendency to flow, and crazing is usually present, although crazing increases toward the low silica and low alumina corner.

The low alumina and low silica corner produces crazing, pin holes, immaturity and where SnO_2 is used it leaves the SnO_2 apparently undissolved. (See result of DM and DN groups).

It was noted that in group DK the glazes were clear even in the low silica and low alumina corner when the SnO_2 was present, but the lowest alumina molecules were .3 whereas in groups DM and DN the molecules were .25, which might account for it. This segregation might also be attributed to a slight reducing atmosphere in the kiln. When the finishing fire was forced by the use of strong air pressure, this same segregation of particles was apparent. When the finishing burn was carried on with low air pressure, giving perfect oxidizing atmosphere, the segregation was not apparent.

The high silica and high alumina corner produces beading, immaturity and waviness although no crazing is observed.

The low alumina-high silica corner produces fair, bright glazes, but in nearly every case the glazes show waviness becoming rougher toward the high silica and high alumina corner.

Group R shows that the use of .1 molecule of PbO and low spar content produces good glazes and mats between .2 to .35 Al_2O_3 and 1.5 to 2.25 SiO_2. A glaze having this RO seems to cover the body well, having less viscosity than a glaze without lead.

Group S shows that low spar content, high ZnO and MgO (.41 molecules together) produce a very hard viscous glaze which requires more heat than cone 3. The only glaze which fuses has .35 Al_2O_3 and 2. SiO_2.

It will be noted that group DK which has a similar RO including .05 SnO_2, produces at cone 4 about the same texture glaze as .35 Al_2O_3 and 2.6 SiO_2, although with .35 Al_2O_3 and 2.0 SiO_2 it is crazed and matted. It evidently takes a trifle more SiO_2 to dissolve the SnO_2.

The average ratio between silica and alumina producing mats and semi-mats is given below. These glazes are selected at random from each group, but illustrate that where no SnO_2 is present a good mat is produced with a ratio of 1 to 5, whereas with .05 SnO_2 the ratio changes to 1 to 6, and where .12 SnO_2 is used the ratio becomes 1 to 7.

Glaze Numbers.	*Al_2O_3*	*SiO_2*	*SnO_2*	*Ratio*
X No. 12.....	.44	2.06	0	1 to 4.6
AE No. 6.....	.475	2.40	.02	1 to 5.1
CA No. 9.....	.45	2.10	.04	1 to 5.
DK No. 13.....	.45	2.6	.05	1 to 6.5
DM No. 3.....	.45	2.75	.05	1 to 6.
CR No. 1.....	.28	2.00	.12	1 to 7.2

Group T shows that high K_2O produces good bright glazes between .4 to .5 Al_2O_3 and 2.4 to 2.9 SiO_2.

Group X shows that with high content of MgO and ZnO and low K_2O, the range of glazes lie only between .4 to .48 Al_2O_3 and 2.06 to 2.68 SiO_2.

Group AE shows that with fairly high K_2O and a trifle less ZnO and MgO than the X group, the fusing point is lowered; we have a range of from .4 to .5 Al_2O_3 and 2.4 and 2.9 SiO_2 and much whiter and better glazes than in group X.

Group AH shows that, with a very low content of K_2O, even the addition of .20 PbO gives us only a range of from .312 Al_2O_3 to .35 Al_2O_3 and from 1.5 to 2.25 SiO_2.

Group BO using high K_2O gives us a range from .3 to .4 Al_2O_3 and from 2.4 to 2.9 SiO_2.

Group CA gives us a narrow range on account of the K_2O content being low but produces fair glazes between .4 and .5 Al_2O_3 and 2.1 SiO_2.

Group CF containing very low K_2O together with low SiO_2 produces glazes between .3 to .4 Al_2O_3 and 1.6 to 2.2 SiO_2.

Group CR ranges between .24 to .28 Al_2O_3 and .2 to 2.3 SiO_2 evidently on account of high content of SnO_2.

Group DK having a medium amount of K_2O produces a fairly good range of from .3 to .45 Al_2O_3 to 2.6 to 2.9 SiO_2.

Group DM having high content of CaO gives us a range of from .25 to .4 Al_2O_3 and 2.375 to 3.125 SiO_2.

Group DN being harder than DM group on account of the high ZnO and MgO content, gives us only a range of from .25 to .35 Al_2O_3 and 2.75 to 3.1 SiO_2. Brandywine spar was used.

Comparing this group with the AE group we note that the latter contains more K_2O, less ZnO and less CaO, and the range is .4 to .5 Al_2O_3 and 2.4 to 2.9 SiO_2. Norwegian spar was used.

From this comparison, we infer that for good mats use a rather hard spar and introduce at least .35 to .38 molecules K_2O, using less than .35 molecules of ZnO and fairly low CaO. For clear glazes, using the BO group for example, try .3 K_2O, .3 CaO, .3 ZnO, .05 BaO and .05 MgO.

Regarding the ratio between the RO, alumina and silica, we have picked five good mat glazes from the groups, each with a different RO, and it may readily be seen by comparison that the ratio of silica to alumina and also the ratio of silica and alumina to the RO is by no means similar. (Mat glazes as follows:)

DK — 13

$$\left.\begin{matrix} .25\ K_2O \\ .36\ ZnO \\ .06\ MgO \\ .33\ CaO \end{matrix}\right\} .4\ Al_2O_3 \left\{\begin{matrix} 2.6\ SiO_2 \\ .05\ SnO_2 \end{matrix}\right.$$

AE — 1

$$\left.\begin{matrix}.38\ K_2O\\ .35\ ZnO\\ .17\ CaO\\ .05\ MgO\\ .05\ BaO\end{matrix}\right\} .5\ Al_2O_3 \left\{\begin{matrix}2.4\ SiO_2\\ .02\ SnO_2\end{matrix}\right.$$

X-12

$$\left.\begin{matrix}.02\ K_2O\\ .15\ MgO\\ .30\ ZnO\\ .35\ CaO\end{matrix}\right\} .44\ Al_2O_3 \left\{\ 2.06\ SiO_2\right.$$

DM-8

$$\left.\begin{matrix}.02\ K_2O\\ .05\ CaO\\ .03\ ZnO\end{matrix}\right\} .40\ Al_2O_3 \left\{\begin{matrix}2.75\ SiO_2\\ .05\ SnO_2\end{matrix}\right.$$

CA — 14

$$\left.\begin{matrix}.25\ K_2O\\ .34\ CaO\\ .33\ ZnO\\ .05\ MgO\\ .03\ BaO\end{matrix}\right\} .4\ Al_2O_3 \left\{\begin{matrix}1.8\ SiO_2\\ .04\ SnO_2\end{matrix}\right.$$

It appears that whatever RO is selected one can find a combination of silica and alumina which will be best suited to it although the temperature may have to be raised a trifle for a very hard RO.

The materials, duration of burn and time of cooling decidedly influence the results, and if materials other than those mentioned in this paper are used, it will be necessary to make up a new group as results will differ widely.

NOTES ON BARIUM GLASSES

BY A. E. WILLIAMS AND S. F. COX

The value of barium and the properties which it imparts to glasses presents a subject of sufficient interest to make experimental work desirable. The following work was planned to gather information regarding the various physical and chemical properties of a series of glasses and also the specific properties imparted by adding barium in varying amounts to a highly alkaline glass.

The literature regarding the use of barium in glass is rather meager and consists in most cases of an expression of opinion. It has been reviewed by Silverman[1] in a recent paper, and presents the ideas of various writers regarding the value and possible use of barium.

As a summation of the same, one may conclude that barium acts as might be expected from its position in the periodic table. It will replace lime or lead to any extent and decrease solubility in the same manner as lime or lead, although possibly not to the same extent. In addition, barium is said to impart somewhat more brilliancy and strength than lime, and to flow well in molding, in other words, produce a more plastic glass. This is caused possibly by a slower rate of change of viscosity with temperature.

The experimental work was planned, moreover, with the idea of producing a highly insoluble glass having reasonably easy working properties. It was desired, also, to determine the specific properties of a potash barium glass as compared with a soda barium glass, the properties of which the literature discusses in a more or less brief manner. A group of 42 glasses was made consisting of glasses varying in composition[2] from

[1] The Use of Barium Compounds in Glass, Alexander Silverman. *Jour. Soc. of Chem. Ind.*, Apr. 30, 1915.

[2] See Tables I, II, and III, showing composition.

that comparable to a Jena soft crown to a dense barium crown, thus giving glasses of a wide variation in solubility and working properties.

TABLE I. MOLECULAR FORMULA OF GLASSES

No.	SiO_2	Na_2O	BaO	ZnO	K_2O
1	2.5	.5	0	.1	.4
2	2.5	.4	.1	.1	.4
3	2.5	.3	.2	.1	.4
4	2.5	.2	.3	.1	.4
5	2.5	.1	.4	.1	.4
6	2.5	.0	.5	.1	.4
7	3.0	.5	.0	.1	.4
8	3.0	.4	.1	.1	.4
9	3.0	.3	.2	.1	.4
10	3.0	.2	.3	.1	.4
11	3.0	.1	.4	.1	.4
12	3.0	.0	.5	.1	.4
13	3.5	.5	.0	.1	.4
14	3.5	.4	.1	.1	.4
15	3.5	.3	.2	.1	.4
16	3.5	.2	.3	.1	.4
17	3.5	.1	.4	.1	.4
18	3.5	.0	.5	.1	.4
19	4.0	.5	.0	.1	.4
20	4.0	.4	.1	.1	.4
21	4.0	.3	.2	.1	.4
22	4.0	.2	.3	.1	.4
23	4.0	.1	.4	.1	.4
24	4.0	.0	.5	.1	.4
25	4.5	.5	.0	.1	.4
26	4.5	.4	.1	.1	.4
27	4.5	.3	.2	.1	.4
28	4.5	.2	.3	.1	.4
29	4.5	.1	.4	.1	.4
30	4.5	.0	.5	.1	.4
31	5.0	.5	.0	.1	.4
32	5.0	.4	.1	.1	.4
33	5.0	.3	.2	.1	.4
34	5.0	.2	.3	.1	.4
35	5.0	.1	.4	.1	.4
36	5.0	.0	.5	.1	.4
37	5.5	.5	.0	.1	.4
38	5.5	.4	.1	.1	.4
39	5.5	.3	.2	.1	.4
40	5.5	.2	.3	.1	.4
41	5.5	.1	.4	.1	.4
42	5.5	.0	.5	.1	.4

The raw materials used in preparing the batches for these glasses consisted of ground potters flint, commercial zinc oxide, 58 percent soda ash, commercial potash, and precipitated barium carbonate.

A 300 gram batch was made which, after being weighed out, was thoroughly mixed by passing through a 20 mesh screen several times. The melting was done in a Steel Harvey tilting furnace, heated with fuel oil. Four batches were melted at once in Battersea crucibles. The process of melting was to heat the crucibles to about 1200° C., pour half of the batch in each crucible, raise the furnace to about 1400° C. to fuse the batch in the crucible then add the remainder of the batch and proceed with the melting. In melting, the furnace was raised to about 1450° C. for the softer glasses and to about 1550° C. for the harder glasses. The furnace was held at this temperature for six hours when the glasses were, in nearly all cases, well melted and free from bubbles. When melting was completed, the glasses were poured into a hot iron mold and removed as soon as solid to a tempering oven. Here, they were cooled from a temperature of 500° C. to atmospheric temperature in about 12 hours.

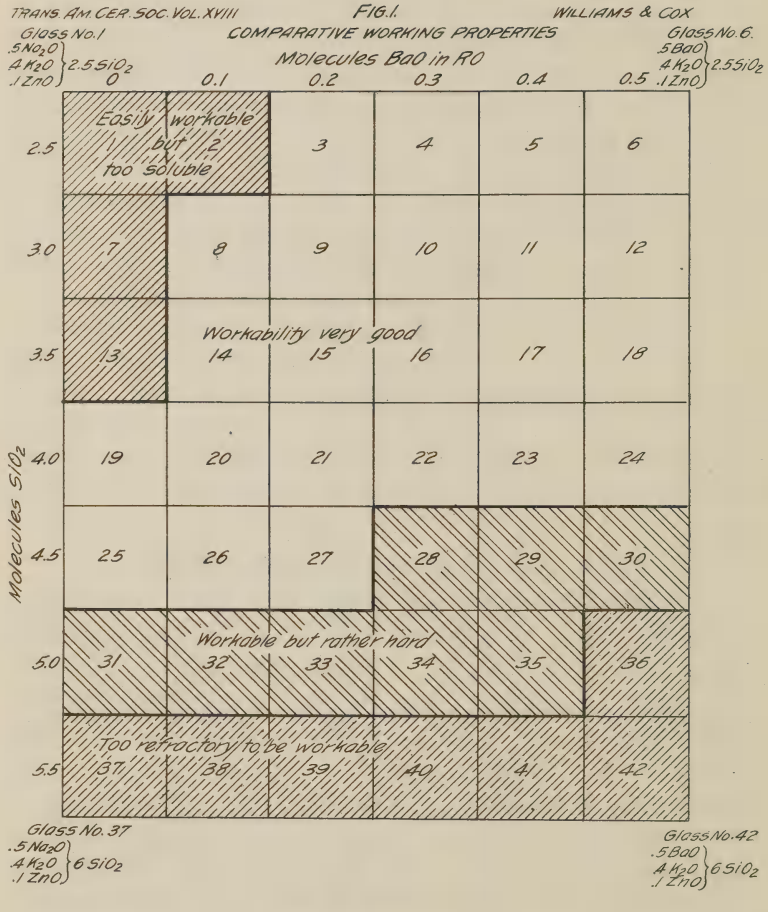
RESULTS OF EXPERIMENTAL WORK

The properties which were determined either by observation, or calculation are: 1st, a relative idea of the working properties, such as ease of melting and molding, comparative appearance with respect to brilliancy; 2nd, density; 3rd, coefficient of expansion; 4th, solubility; 5th, devitrification properties; 6th, softening point; 7th, viscosity range.

Working Properties. The potash barium glasses showed a decided increase in brilliancy with increase in barium. Glasses which contained no soda and five-tenths of a molecule of barium appeared to have a comparatively better lustre than some high quality cut glass goods.

The glasses were increasingly more difficult to melt as they approached the high silica-high barium corner of the diagram, barium increasing the refractoriness much more than silica. Glasses Nos. 30 and 36 to 42 were very hard to melt and at

1550° C. were much too viscous to pour from the crucible into the mold. Of these eight glasses, Nos. 36 and 42 were not free from bubbles when melted for 6 hours at 1600° C. The balance



of the glasses were not difficult to melt and became fluid enough to flow easily within the range of temperature mentioned above. Those high in barium poured best and remained viscous or plastic like a lead glass, apparently longer than the high soda potash

TABLE II. PERCENT COMPOSITION OF GLASSES

NO.	SiO_2	Na_2O	BaO	ZnO	K_2O
	percent	*percent*	*percent*	*percent*	*percent*
1	66.3	13.7	.....	3.6	16.5
2	63.6	10.5	6.5	3.4	16.0
3	61.4	7.6	12.5	3.3	15.4
4	59.0	4.9	18.0	3.2	14.8
5	57.1	2.3	23.3	3.1	14.3
6	55.2	.....	28.0	3.0	13.8
7	70.0	12.1	.....	3.2	14.6
8	67.7	9.3	5.8	3.0	14.2
9	65.4	6.8	11.1	2.9	13.7
10	63.4	4.4	16.2	2.8	13.2
11	61.4	2.6	20.9	2.8	12.8
12	59.4	.....	25.3	2.7	12.4
13	73.3	10.8	.....	2.8	13.1
14	71.1	8.4	5.2	2.7	12.7
15	69.0	6.1	10.0	2.7	12.6
16	67.0	3.9	14.6	2.6	12.0
17	65.0	1.9	19.0	2.5	11.6
18	63.3	.....	23.0	2.4	11.3
19	75.7	9.8	.....	2.6	11.9
20	73.7	7.6	4.7	2.5	11.5
21	71.7	5.5	9.1	2.4	11.2
22	69.8	3.6	13.3	2.4	11.0
23	68.0	1.7	17.3	2.3	10.7
24	66.2	.....	21.0	2.2	10.4
25	78.0	8.9	.....	2.3	10.8
26	76.0	5.6	4.3	2.3	10.6
27	74.0	5.1	8.4	2.2	10.3
28	72.2	3.3	12.2	2.2	10.1
29	70.5	1.6	16.0	2.1	9.8
30	68.9	.....	19.5	2.1	9.6
31	79.6	8.2	.....	2.2	10.0
32	77.8	6.4	4.0	2.1	9.8
33	76.0	4.7	7.7	2.0	9.5
34	74.4	3.1	11.3	2.0	9.3
35	72.7	1.5	14.8	1.9	9.1
36	71.1	.....	18.3	1.9	8.9
37	81.3	7.6	.....	2.0	9.2
38	79.4	6.0	3.7	1.9	9.0
39	77.8	4.4	7.2	1.9	8.8
40	76.3	2.9	10.5	1.8	8.7
41	74.6	1.4	13.7	1.8	8.5
42	73.0	.....	16.9	1.8	8.3

TABLE III. BATCH WEIGHT OF GLASSES

NO.	SiO_2	Na_2CO_3	$BaCO_3$	K_2CO_3	ZnO
1	150	70.5	.....	75.2	8.1
2	150	56.4	19.7	75.2	8.1
3	150	42.3	39.5	75.2	8.1
4	150	28.2	57.2	75.2	8.1
5	150	14.1	79.0	75.2	8.1
6	150	.....	98.7	75.2	8.1
7	180	70.5	.....	75.2	8.1
8	180	56.4	19.7	75.2	8.1
9	180	42.3	39.5	75.2	8.1
10	180	28.2	57.2	75.2	8.1
11	180	14.1	79.0	75.2	8.1
12	180	.....	98.7	75.2	8.1
13	210	70.5	.....	75.2	8.1
14	210	56.4	19.7	75.2	8.1
15	210	42.3	39.5	75.2	8.1
16	210	28.2	57.2	75.2	8.1
17	210	14.1	79.0	75.2	8.1
18	210	.....	98.7	75.2	8.1
19	240	70.5	.....	75.2	8.1
20	240	56.4	19.7	75.2	8.1
21	240	42.3	39.5	75.2	8.1
22	240	28.2	57.2	75.2	8.1
23	240	14.1	79.0	75.2	8.1
24	240	.....	98.7	75.2	8.1
25	270	70.5	.....	75.2	8.1
26	270	56.4	19.7	75.2	8.1
27	270	42.3	39.5	75.2	8.1
28	270	28.2	57.2	75.2	8.1
29	270	14.1	79.0	75.2	8.1
30	270	.....	98.7	75.2	8.1
31	300	70.5	.....	75.2	8.1
32	300	56.4	19.7	75.2	8.1
33	300	42.3	39.5	75.2	8.1
34	300	28.2	57.2	75.2	8.1
35	300	14.1	79.0	75.2	8.1
36	300	.....	98.7	75.2	8.1
37	330	70.5	.....	75.2	8.1
38	330	56.4	19.7	75.2	8.1
39	330	42.3	39.5	75.2	8.1
40	330	28.2	57.2	75.2	8.1
41	330	14.1	79.0	75.2	8.1
42	330	.....	98.7	75.2	8.1

glasses. The apparent comparative working properties of the glasses are shown on the diagram Fig. I.

Density. Due to the work of Winkleman and Schott[3], factors are available from which the density of glasses may be calculated. The formula and factors used in the calculation of density are given herewith.

$$\frac{A_1}{Z_1} + \frac{A_2}{Z_2} + \frac{A_3}{Z_3} = \frac{100}{S}$$

A = percent of oxide in glass.
Z = factor.
S = Density.

Factors — $SiO_2 = 2.3$ — $ZnO = 5.9$ — $BaO = 7.0$ — $Na_2O = 2.6$ — $K_2O = 2.8$.

The density of the glasses made was calculated from the above formula and are given in Table V. For purpose of check and comparison the densities were also carefully determined by the suspension method. Fragments of glass weighing about 5 grams and carefully chosen, so as to be free from bubbles were used for this purpose.

A comparison of the two sets of data[4] does not show any particular direction for an increasing error except that the error is generally greater in the glasses containing .5 mol. of BaO rather than the glasses containing .5 mol. Na_2O. There is, however, nothing which would indicate the incorrectness of any one factor.

As the table shows[5], high density is obtained in the low silica, high barium corner of the field and low density in the opposite corner. As would be expected from the factors increasing SiO_2 decreases density and increasing BaO increases density, the densest glass (No. 6) showing a density of 2.990 and the lightest glass (No. 37) having a density of 2.385.

Coefficient of Expansion. The coefficient of expansion of this group of glasses was calculated by use of Winkleman and Schott's factors.[6] The following factors were used which when

[3] *Jena Glass*, Hovesetadt, p. 148.
[4] Tables IV and V.
[5] Tables IV and V.

multiplied by the percent of oxide used give by addition the value of the cubic expansibility 3a in mm. per degree C. multiplied by 10^7.

Na_2O — 10.0, K_2O — 8.5, BaO — 3.0, ZnO — 1.8, SiO_2 — 0.8.

This gives a range of expansibility approaching that of metal to an expansibility nearly as low as is reported for glasses. The maximum linear expansion (a) being .0000112 as compared with the expansion of iron .000013. The minimum expansibility calculated, (glass No. 42) = .00000609. Sullivan[7] reports a new glass having an expansibility of .0000032. Hovestadt[8] reports a Jena zinc borate glass having an expansibility of .0000037.

The expansibilities shown,[9] however, would be considered quite low for a glass free from boric acid.

Solubility. The best means of determining the solubility of glass in water is open to discussion. It seemed to the writers, however, that the great activity of water at high temperatures as a solvent, made the method used quite desirable for determining the actual solubility of these glasses. E. C. Sullivan[10] in a recent article describes a method quite similar as the most accurate one. Following is a description of the method used: Glass plates were prepared having a size closely approximating the dimensions 0.35 cm. by 4.0 cm. by 8 cm., the four thin edges being ground on a wheel.

These pieces were accurately measured with calipers and their areas calculated. The pieces were soaked in distilled water for 48 hours, after a thorough cleansing with alkali water. After the 48 hours soaking, they were placed in glazed porcelain casseroles, a definite amount of distilled water (200 cc.) was added and kept at a temperature of 80° C. in an electric oven for 24 hours. A practically constant volume of water was kept in the casseroles by keeping the atmosphere of the oven saturated. This latter was necessary to control the concentration of the solution. The solution was then poured into a platinum evaporating dish, the glass and casserole washed with 50 cc. of distilled water

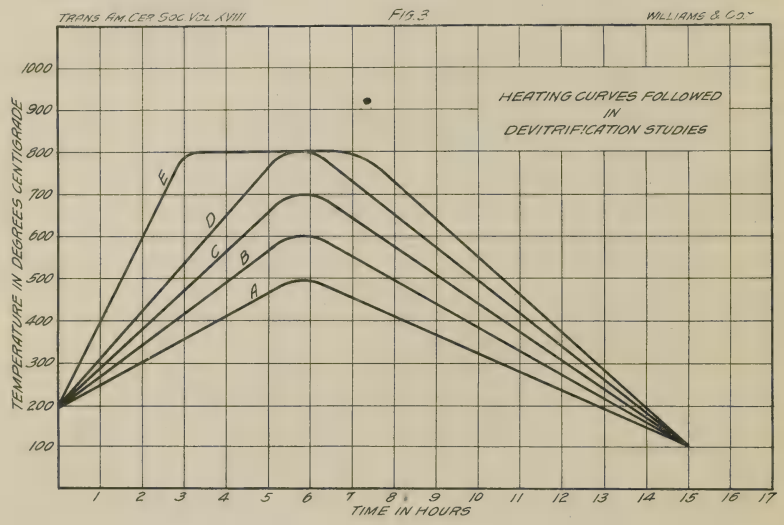
[6] *Jena Glass*, Hovestadt, p. 217.
[7] *Development of Low Expansion Glasses.* A. C. Sullivan, *Jour. Ind. and Eng. Chem.*, May, 1916, p. 399.
[8] *Jena Glass*, Hovestadt, p. 216.
[9] Table VI.
[10] Development of Low Expansion Glasses, E. C. Sullivan, *Jour of Ind. and Eng. Chem.*, May, 1916, p. 399.

Group I - Solubility above 1.0 gms. X 10^{-5} per sq. M.M. All glasses showed disintegration
Group II - Solubility between 1.0 gms. X 10^{-5} per sq. M.M. No disintegration apparent
Group III - Solubility between 0.1 $^{-5}$ gms. per sq. M.M. and 0

which was also added to the solution, and the solution evaporated to dryness and weighed.

Several blank determinations were made on the distilled water and casseroles, and the blank determined subtracted from the total weight.

A few determinations of solubility were run using a definite weight of ground glass of a definite size of grain, the relative solubility of the various glasses showed practically no different than shows by the method used.



The solubility of these glasses is shown in Table VII, expressed in grams x 10^{-5} per square millimeter of surface exposed to the solvent. Their physical appearance is also described in Table VIII.

Figure 2 shows the glasses classified into three groups, those of excessive solubility, that is, glasses too soluble to be practical for any ordinary purpose are classed in Group 1 having a solubility greater than 1.0 gm. x 10^{-5} per sq. mm. surface. The glasses in this group all showed more or less disintegration in the solubility test, some showing only a few chips or spalls, and

TABLE IV. OBSERVED DENSITY OF GLASSES

1	2.456	15	2.592	29	2.670
2	2.493	16	2.657	30	2.692
3	2.681	17	2.750	31	2.411
4	2.758	18	2.815	32	2.483
5	2.863	19	2.427	33	2.495
6	2.985	20	2.500	34	2.543
7	2.478	21	2.559	35	2.615
8	2.567	22	2.622	36	2.656
9	2.650	23	2.693	37	2.379
10	2.720	24	2.745	38	2.439
11	2.799	25	2.421	39	2.499
12	2.865	26	2.480	40	2.532
13	2.451	27	2.541	41	2.573
14	2.533	28	2.592	42	2.630

TABLE V. CALCULATED DENSITY OF GLASSES.

1	2.462	15	2.581	29	2.675
2	2.548	16	2.668	30	2.738
3	2.668	17	2.757	31	2.398
4	2.781	18	2.838	32	2.458
5	2.885	19	2.417	33	2.520
6	2.990	20	2.489	34	2.575
7	2.448	21	2.567	35	2.639
8	2.528	22	2.615	36	2.700
9	2.630	23	2.711	37	2.385
10	2.720	24	2.786	38	2.444
11	2.800	25	2.405	39	2.501
12	2.912	26	2.453	40	2.550
13	2.428	27	2.540	41	2.615
14	2.513	28	2.604	42	2.672

TABLE VI. CUBICAL EXPANSION — 3a (CALCULATED)

Expressed as 10^7 times the cubical expansibility per mm. per degree C. Interval 0-100°C.

1	336.83	15	258.06	29	207.38
2	317.52	16	243.08	30	198.98
3	309.59	17	231.10	31	234.66
4	287.96	18	219.94	32	225.28
5	266.68	19	264.48	33	215.20
6	251.10	20	251.40	34	207.00
7	316.96	21	239.24	35	198.42
8	290.80	22	229.62	36	190.82
9	275.37	23	223.24	37	222.80
10	260.54	24	208.36	38	214.52
11	251.69	25	247.34	39	206.12
12	233.76	26	235.84	40	198.74
13	283.09	27	226.96	41	190.44
14	269.36	28	217.36	42	182.94

others being so badly disintegrated that they may be picked to pieces. Special attention may be called to glass No. 2 showing an extremely high solubility. Several check determinations were made on this glass from the same melt and from different melts. This glass also shows a marked difference in its working properties as compared with the others, it being an exceptionally soft glass.

In Group 2 on Fig. 2 the solubility of the glasses are all low enough for any purpose except chemical glass ware, the solubility of this group being between 1.0 grams and 0.1 grams x 10^{-5} per sq. mm. These glasses showed no signs of disintegration in the solubility test.

The glasses of group three, having a solubility below 0.1 grams x 10^{-5} per sq. mm. of surface, are practically insoluble many of them showing a solubility within the variation of the blank. In this group there are also many apparently easily workable glasses as Nos. 11, 12, 17, 18, 22, 23, 24, 28, 29 and 30.

It will be noted, first, that increasing BaO shows a much more marked decrease in solubility than increasing silica; second, that a glass containing only two and one-half molecules of SiO_2 is practically insoluble with five-tenths molecule of BaO; third, that at least three-tenths molecule of BaO is required to produce a highly insoluble glass.

Devitrification. The method of determining the devitrification properties of these glasses was to heat a sample of each glass in a muffle furnace according to the temperature curves shown on Fig. 3. The samples were placed on an asbestos board in the muffle furnace and heated and cooled according to curve marked A. They were then examined and reheated according to curve B, etc. By using this method of heating up to the glass, the relative tendency of the various glasses to devitrify would make itself evident.

None of these glasses showed any indication of devitrification in pouring or annealing. In the heating of the glasses to produce devitrification, not any of them showed complete devitrification or become opalescent throughout the glass. Fifteen glasses showed surface devitrification after being reheated to 700° C. This increased in the two heat treatments following at

800° C., but at no time was the devitrification more than a superficial film, nor did it show up on any other glasses except those which showed this property at 700° C. All glasses except those

containing 5 mol. barium were heated beyond the deformation point.

The glasses which showed surface devitrification are shown in Fig. 4, which indicates that barium in these soda potash zinc

TABLE VII. SOLUBILITIES

Expressed in Grams $\times 10^{-5}$ per sq. mm. of surface exposed to solvent.

1	8.194	15	.503	29	.0859
2	109.70	16	.0173	30	.0252
3	1.207	17	.0620	31	1.348
4	.4609	18	.000	32	.2522
5	.1405	19	5.688	33	.2463
6	.0396	20	.857	34	.0171
7	18.390	21	.274	35	.0176
8	1.470	22	.0438	36	.0102
9	1.569	23	.0339	37	.4909
10	.1418	24	.0500	38	.1958
11	.0180	25	3.497	39	.1488
12	.0280	26	.6276	40	.0824
13	7.708	27	.0707	41	.0340
14	1.283	28	.0882	42	.0683

TABLE VIII. APPEARANCE OF SAMPLES AFTER SOLUBILITY TEST

1. Surface very badly spalled and disintegrated, not cracked thru much.
2. Surface slightly worse than No. 1, more deep cracks. The water about this sample was evaporated to dryness.
3. Surface shows a considerable number of spalls and small cracks. Very much improved over No. 1 and No. 2.
4. Does not show disintegration nor spalling off like Nos. 1, 2 and 3. Water was evaporated to dryness about this glass and it cracked badly.
5. No apparent disintegration.
6. No apparent disintegration.
7. Surface not spalled and cracked, Much like Nos. 1, 2 and 3, but completely covered with white film of soft granular material, giving the glass a streaked milky appearance.
8. Only about a dozen small spots where glass has spalled off. No scum on surface.
9. Spalled about same as No. 8.
10. Very clear glass, no apparent disintegration.
11. No apparent disintegration.
12. No apparent disintegration
13. Surface all white, spalled in a few places, apparently not any better than Nos 1, 2, 3 and 7.
14. Just three small chipped or spalled spots.
15. Apparently no disintegration.
16. Apparently no disintegration.
17. Glass cracked in a couple of places.
18. Apparently no disintegration.

19. Spalled considerably, and has a faint white scum. Considerably better than Nos. 1, 2, 3, 7 and 13.
20. About a dozen chipped and spalled spots.
21. No apparent disintegration.
22. No apparent disintegration.
23. One sample surface badly chipped, disintegrated; evaporated to dryness. Another melt made.
24. No apparent disintegration.
25. Surface badly chipped and spalled, thin film of white scum, but less than No. 19.
26. No apparent disintegration.
27. No apparent disintegration.
28. No apparent disintegration.
29. No apparent disintegration.
30. No apparent disintegration.
31. Surface covered with superficial cracks and shows a few spalled spots, a slight film of scum in spots. Much better than No. 25.
32. No apparent disintegration.
33. No apparent disintegration.
34. Piece cracked thru, from pouring cold water on hot glass. Water evaporated to dryness during solubility test.
35. No apparent disintegration.
36. No apparent disintegration.
37. No apparent disintegration.
38. No apparent disintegration.
39. No apparent disintegration.
40. No apparent disintegration.
41. No apparent disintegration.
42. No apparent disintegration.

barium glasses does not increase devitrification, in fact the tendency to devitrify was evident only in the high silica corner. A sample of the glass which showed surface devitrification was examined by Mr. A. A. Klein, of the Bureau of Standards, who reported that he found the typical aborescent structure in the material which was apparently devitrified, but that the individual crystals were so minute that they appeared isotropic and could not be identified.

Although the crystalline material was not analyzed, this surface devitrification seems to corroborate Brockbank's[11] explanation of surface devitrification, being due to the volatilization of potassium or sodium oxide from the glass and producing a sur-

[11] *Trans. Amer. Cer. Soc.*, Vol. XV, p. 600

face layer of a silicate containing a very high percentage of SiO_2.

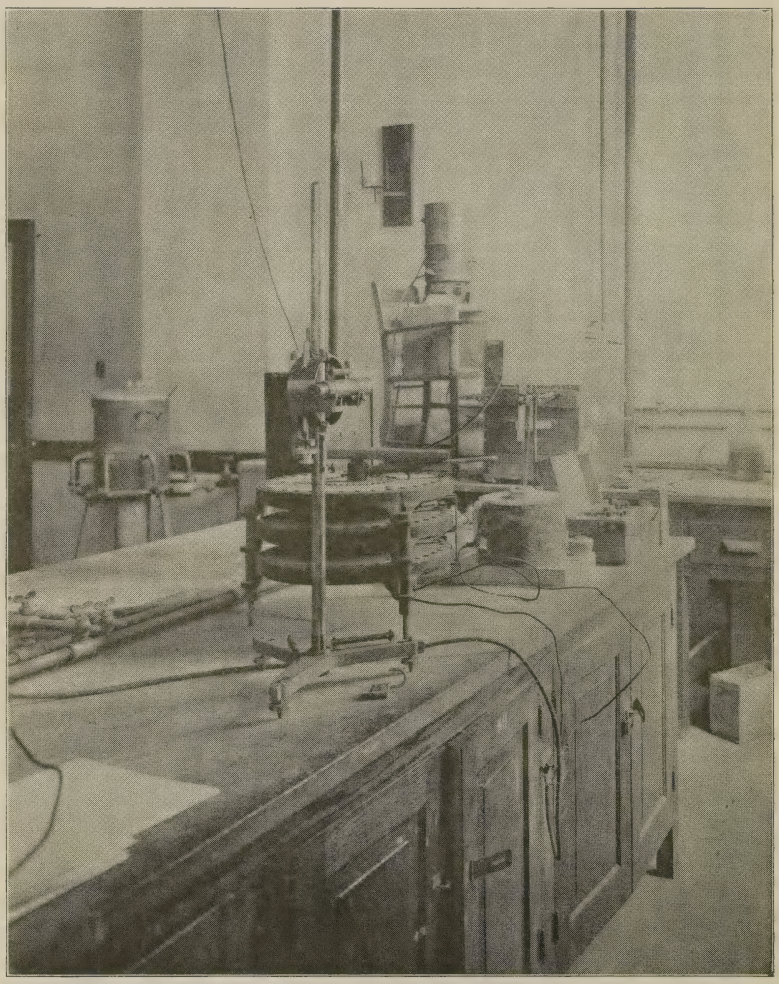
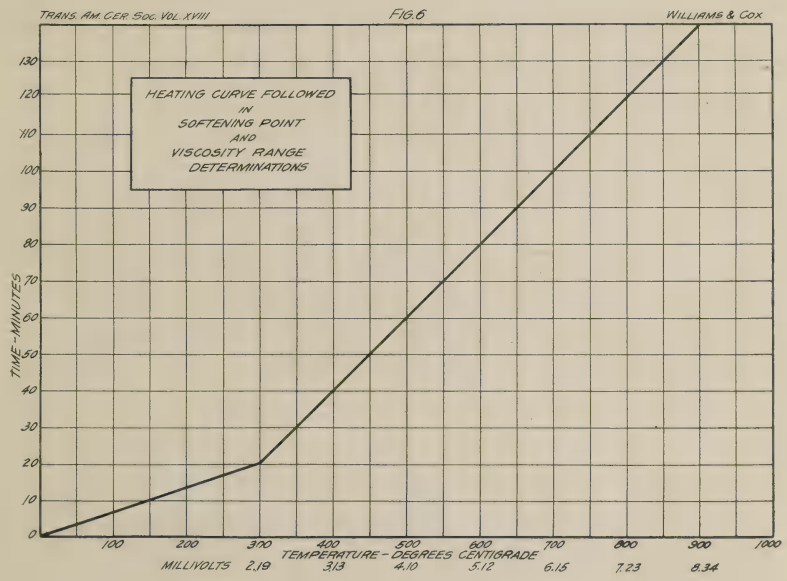


Fig. 5.

Softening Points. Various methods for the determination of the softening point of a glass have been used. Owing to no standard method of measuring this factor, the results

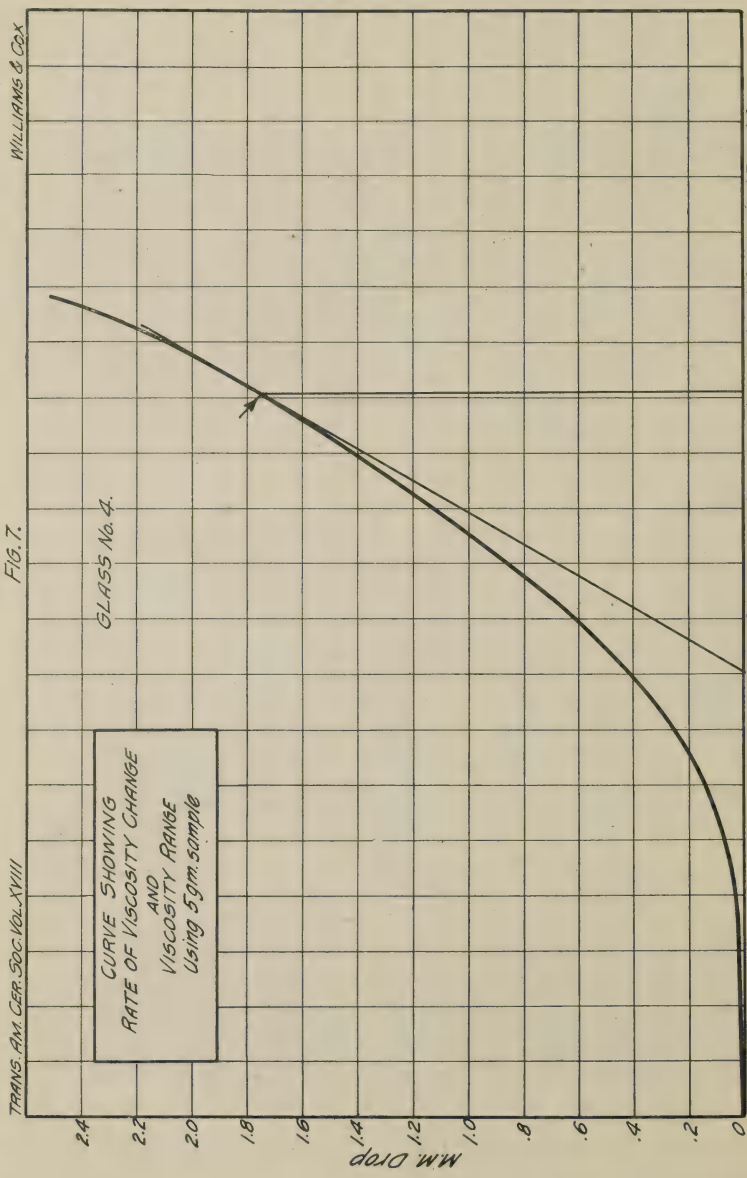
will be of value only in comparing the relative softening points of the glasses made.

The method of determining the softening point was by noting the temperature at which a weighted rod begins to penetrate the glass surface. This was chosen for the further purpose of noting the rate of change of viscosity of the glasses, with increasing temperature, by determining the rate at which the rod is sinking.



A photograph of the apparatus used is shown in Fig. 5, and a description of the same is as follows:—

Five grams and later, 10 grams of glass, were softened into a solid mass in a small Battersea crucible about 1½ in. high and 1 in. in diameter at the top. This was placed in a small electric furnace heated by a platinum coil. A fire clay stopper was placed over the furnace opening, thru which a thermo-couple was passed, the junction resting on the surface of the glass. Thru another hole in the center of the cork a platinum rod was passed also resting on the surface of the glass. The upper end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TRANS. AM. CER. SOC. VOL. XVIII
FIG. 7.
GLASS No. 4.
CURVE SHOWING
RATE OF VISCOSITY CHANGE
AND
VISCOSITY RANGE
Using 5gm. sample
mm drop
TEMPERATURE DIFFERENCE IN MILLIVOLTS

of the rod was weighted with lead. A copper wire about 8 inches long, having a pointer bent at right angles, fitted into a hole on top of the platinum rod. This wire was supported by passing it thru a glass tube. The entire rod was then set so as to stand perpendicular on the glass surface, and weighed 15.06 gms. The diameter of the platinum rod equaled 1.71 mm., thus giving a weight of 6.39 gms. per sq. mm.

When the rod was properly set on the glass a micrometer telescope was focused on the pointer, the height read, and the drop of the rod measured by lowering the telescope.

The furnace temperature was raised at a constant rate throughout all experiments. Figure 6 shows the heating curve followed.

With increasing temperature, the expansion of the rod showed a slight rise. The rod then remained stationary for a short period, and finally began to drop. The first indication of the rod dropping was called the softening point.

Using a five gram sample of glass, softening points were determined on glasses Nos. 1 to 18 inclusive.

The results are shown on Table VIII, along with check determinations on several of the samples.

A gradual increase in the refractoriness of the glass with increasing barium and silica is shown, barium hardening the glass considerably more than the silica.

A second series of determinations were run using ten grams of glass rather than five. The softening points were determined on glasses Nos. 1 to 7 inclusive, and 36 to 42 inclusive. In addition, the softening points of a flint glass, a crown glass, an opal glass, a zinc glass, and a commercial plate glass were determined for purpose of comparison. The following are the compositions of the above mentioned glasses:—

A. Flint glass

$$\left.\begin{matrix} .35\ K_2O \\ .2\ Na_2O \\ .45CaO \end{matrix}\right\} \quad 3\ SiO_2$$

B. Crown glass

$$\left.\begin{matrix}.34\ K_2O \\ .46\ CaO \\ .38\ PbO\end{matrix}\right\}\ 3\ SiO_2$$

Z. Zinc glass

$$\left.\begin{matrix}.24\ K_2O \\ .20\ Na_2O \\ .46\ CaO \\ .10\ ZnO\end{matrix}\right\}\ 3\ SiO_2$$

Opal glass

$$\left.\begin{matrix}.36\ K_2O \\ .52\ Na_2O \\ .12\ PbO\end{matrix}\right\}\ \left\{\begin{matrix}.026\ B_2O_3 \\ .087\ Al_2O_3\end{matrix}\right\}\ 4.4\ SiO_2$$

Commercial plate—composition unknown.

The softening points of these second determinations, shown also on Table IX, are lower than with the five gram samples, and the softening did not proceed so slowly in the beginning. It will be noted that glass No. 42, having silica and high barium, is the most refractory of the experimental group. The extreme refractoriness of the opal glass **O**, and the extreme softness of the lead glass **A** are also worthy of notice.

TABLE IX. SOFTENING POINTS. 5 GRAM SAMPLES

Temperature in Milli-volts

NO.	M. V.	NO.	M. V.	NO.	M. V.
1	4.93	7	4.75	13	4.60
2	4.61	8	5.08	14	4.91
3	5.21	9	5.33	15	5.11
4	5.32	10	5.48	16	5.13
5	5.84	11	5.88	17	5.98
6	6.15	12	6.54	18	6.71

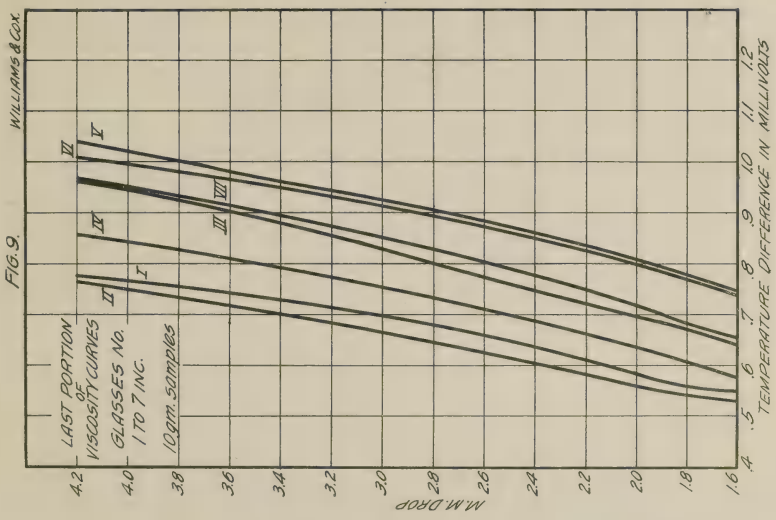
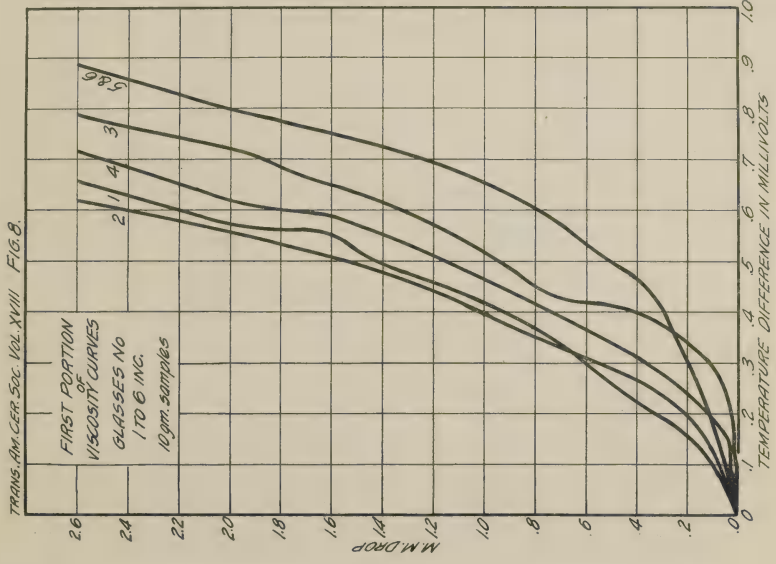


FIG. 9.
WILLIAMS & COX.
LAST PORTION
VISCOSITY CURVES
GLASSES No.
1 TO 7 INC.
10 GM. SAMPLES
I
II
III
IV
V
VI
VII
M.M. DROP
TEMPERATURE DIFFERENCE IN MILLIVOLTS


TRANS. AM. CER. SOC. VOL. XVIII FIG. 8.
FIRST PORTION
OF
VISCOSITY CURVES
GLASSES No
1 TO 6 INC.
10 gm. samples
1
2
3
4
5
6
M.M. DROP
TEMPERATURE DIFFERENCE IN MILLIVOLTS

Determinations Repeated as Checks on Above Values

NO.	FIRST RUN M. V.	SECOND RUN M. V.
6	6.15	6.21
10	5.45	5.48
11	5.51	5.88
12	6.26	6.54
15	5.11	5.05
17	5.98	5.78

10 Gram Samples

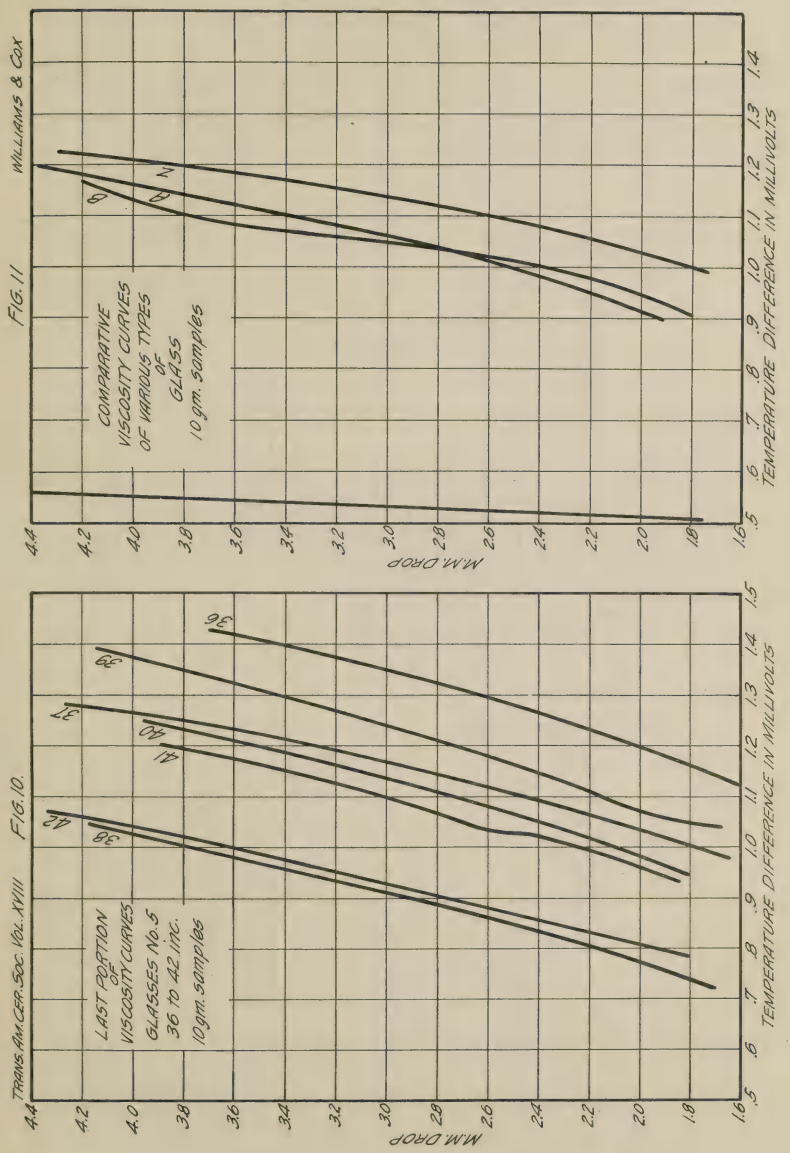
NO.	SOFTENING PT. IN MILLI-VOLTS	NO.	SOFTENING PT. IN MILLI-VOLTS	NO.	SOFTENING PT. IN MILLI-VOLTS	OTHER TYPES GLASS	SOFTENING PT. IN MILLI-VOLTS
1	4.83	7	4.65	37	5.02	O	8.97
2	4.45			38	5.16	A	4.15
3	4.50			39	5.42	Z	6.20
4	4.85			40	5.95	B	6.21
5	5.16			41	6.31	Com.	
6	5.79	36	6.60	42	7.05	Plate	6.06

Viscosity Range. As the workability of a glass is as important to consider as its chemical and physical properties, some measure of this property is desirable. What might be called the viscosity range of a glass or its rate of change in temperature is probably the best criterion of its workability.

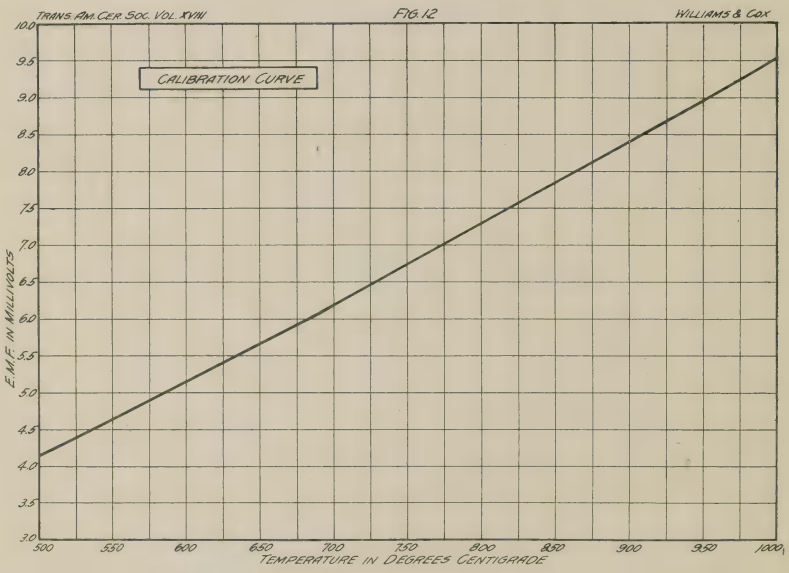
By the measurement of the rate of penetration of a rod, with continually increasing temperature, into a sample of glass, a measure of the rate of change of viscosity may be obtained. The experimental work done to obtain the required data consisted of measuring the drop of the rod and the temperature of the glass every minute, beginning at the softening point.

The first series of results were made on glasses Nos. 1 to 18 for which the softening points are given above. The results of the readings were plotted and curves drawn, are shown on Fig. 7, which is typical of the curves obtained.

The method of interpreting the curve to get a factor which would compare the viscosity range properly, was given much consideration.


WILLIAMS & COX
FIG. 11
COMPARATIVE VISCOSITY CURVES OF VARIOUS TYPES OF GLASS 10 gm. SAMPLES
B
N
A
M.M. DROP
TEMPERATURE DIFFERENCE IN MILLIVOLTS
TRANS. AM. CER. SOC. VOL. XVIII
FIG. 10.
LAST PORTION OF VISCOSITY CURVES GLASSES No. 5 36 TO 42 INC. 10 gm. SAMPLES
42
38
37
39
36
41
40
M.M. DROP
TEMPERATURE DIFFERENCE IN MILLIVOLTS

Two methods of interpreting these curves are shown. The first was to compare the temperature change required to allow the rod to drop to a definite point (2 mm.). This compares, in a way, the rate of change of viscosity, as the greater the difference in temperature between the softening point and the temperature at which a drop of 2 mm. obtained, the slower the rate of change of viscosity.



The second method of interpretation was to compare the temperature difference between the softening point and the point where the tangent of the curve had obtained a slope of 60°, considering that, at this point, each glass had reached the same degree of viscosity, and the curve was close to a constant rate of change of viscosity.

TABLE X

Viscosity Ranges in Milli-Volts for Drop of 2 mm.
5-Gram Sample.

1	1.20	7	1.25	13	1.53
2	1.25	8	1.12	14	1.30
3	1.01	9	1.28	15	1.41
4	1.38	10	1.25	16	1.48
5	1.11	11	1.18	17	1.19
6	1.30	12	1.28	18	1.48

Check Determinations

	FIRST RUN	SECOND RUN
6	1.30	1.30
10	1.25	1.25
11	1.18	1.22
12	1.28	1.30
15	1.41	1.46
17	1.19	1.14

Viscosity Range in Milli-Volts — For Point on Curve Showing Tangent of 60°

5-Gram Sample.

1	1.07	7	1.13	13	1.55
2	1.13	8	1.08	14	1.22
3	.86	9	1.22	15	1.30
4	1.31	10	1.30	16	1.34
5	.95	11	1.14	17	1.10
6	1.24	12	1.24	18	1.14

Rate of Change of Viscosity

10 Gram Sample.
MM. Drop Per 0.1 Milli-Volt Change in Temperature.

NO.	RATE BEGINNING AT 2.0 MM.	RATE BEGINNING AT 2.8 MM.	NO.	RATE BEGINNING AT 2.0 MM.	RATE BEGINNING AT 2.8 MM.	NO.	RATE BEGINNING AT 2.0 MM.	RATE BEGINNING AT 2.8 MM.
	mm.	*mm.*		*mm.*	*mm.*		*mm.*	*mm.*
1	.86	1.40	37	.74	.94	A	.60	1.00
2	1.00	1.10	38	.70	.87	B	.70	1.20
3	.80	.80	39	.57	.74	O	About 6 mm.	About 6 mm.
4	.84	1.02	40	.60	.81	Z	.85	1.40
5	.80	1.02	41	.64	.73			
6	.84	1.10	42	.86	.80			
7	.72	.94	36	.64	.88			

The viscosity range shown by glasses Nos. 1 to 18, using the above methods of determining such a value, is shown on Table X. The two methods give, approximately the same comparative values between any two glasses. The noticeable feature of the results is that the viscosity range does not increase or decrease in a uniform manner with the increase or decrease of any particular ingredient, but on the other hand, seems to increase and decrease alternately. It is quite possible that the viscosity range of glasses would not change uniformly with an increasing quantity of one ingredient, owing to the possibility of different chemical combinations occurring with changes in composition. Several other difficulties were made apparent in plotting the curves, however, which makes the above methods of interpretation unreliable.

The curve shown on Fig. 7, altho being a typical curve, shows a very slow rate of starting. The speed at the softening point of the glass varied considerably and apparently seemed to depend upon other factors than the viscosity of the glass, such as surface tension, level of the glass surface, etc. Therefore, the difference in temperature is not a certain measure of the difference in viscosity range because of the tendency of the dropping of the rod to lag just above the softening point.

The second method is more accurate than the first, as in that case the temperature difference required to obtain a certain definite viscosity is measured, whereas, in the first case no attention is given to the degree of viscosity. Another criticism of this set of experiments, is that the quantity of glass was so small that the rod was approaching close to the bottom of the crucible where it would again be retarded to a certain extent.

In view of the above possibilities of error, in obtaining consistent results, another set of experiments was run using a ten gram sample of glass in place of five grams. The rod in this case was allowed to drop about 4½ mm. rather than only 3 mm., thus giving the portion of the viscosity curve which shows the rate of change of viscosity approximately constant within a short interval (.1 milli-volt or about 10°).

The curves obtained are shown on Figs. 8, 9, 10 and 11. Figure 8 shows the start of the curves which is much more rapid and consistent with the hardness of the glass than the start shown with the smaller sample of glass. Figure 9 shows the balance of the curves for glasses, Nos. 1 to 7. These curves show very little difference in their rate of change of viscosity and also show how nearly the rate of change approaches a constant. Figures 10 shows the same portion of the viscosity curve as Figure 9, but for glasses No. 36 to 42 inclusive. Figure 11 shows the viscosity curves for other types of glasses for comparison.

The rate of change of viscosity was determined by taking the drop of the rod for a change of .1 milli-volt in temperature, using as a starting point a definite amount of drop, 2 mm. in one case and 2.8 mm. in another case. The figures obtained are shown on Table X. The results are more consistent than those obtained by the first two methods and the following general conclusions may be drawn from them.

Replacing sodium with barium decreases the rate of change of viscosity, or in other words, increases the viscosity range, except when all of the sodium is replaced by barium, then a slight decrease of the viscosity range is shown.

Comparing Nos. 1, 7 and 37, and Nos. 6, 36 and 42, there does not seem to be any consistent effect on the viscosity range

due to increasing silica. Comparing the data taken beginning at 2.0 mm. drop with that taken beginning at 2.8 mm. drop, a considerable decrease in the viscosity range or increase in the rate of change is noted with increasing temperature.

The exceptionally rapid rate of change of viscosity of the opal glass is worthy of notice and the slow rate of change of the lead glass is evident.

Fig. 12 is the calibration curve of the thermo-couple used in this work. The softening points in degrees C corresponding to the milli-volts shown on the tables may be taken from this curve.

THE EFFECT OF VARIATION IN THE COMPOSITION OF GROUND COATS FOR SHEET IRON ENAMELS

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The composition of first coats for sheet iron has been a matter of discussion among enamel makers for a long time, especially the effects of composition as regards the use of cobalt, nickel and manganese oxides. The ceramic literature contains as little about this subject as it does about the technical side of most of the enamel industry. It was the writer's intention to determine the effects of these oxides and the percentages of each necessary to produce the best enamel.

With this idea in mind, nine series were outlined as follows:

Series A. Increasing cobalt oxide from .01 to .04 equivalents.

Series B. Increasing nickel oxide from .01 to .05 equiv.

Series C. Increasing cobalt oxide with corresponding decrease in nickel oxide.

Series D. Increasing manganese oxide from .02 to .114 equivalents.

Series E. Increasing cobalt oxide with decrease in manganese oxide.

Series F. Increasing silica.

Series G. Increase of calcium fluoride.

Series H. Decrease of borax.

Series J. Increase of antimony oxide.

A ground coat which was an average of several commercial formulas and approached closely the formula used by Menzel in his work on "The Function of Cobalt in Ground Coats,"[1] was selected as a basis for the above series. This formula, disregarding the cobalt, nickel, and manganese oxides, is as follows:—

[1] *Keramische Rundschau*, 1914, p. 50-52.

$$\left.\begin{matrix} .25\ K_2O \\ .50\ Na_2O \\ .25\ CaO \end{matrix}\right\} \left\{\begin{matrix} .15\ Al_2O_3 \\ .50\ B_2O_3 \end{matrix}\right\} \left\{\begin{matrix} 2.0\ SiO_2 \\ .25\ F_2. \end{matrix}\right.$$

Substitutions were made in the RO group, K_2O, Na_2O, or CaO being replaced in such a way that the change in any one of the above was as small as possible. The change in some cases was naturally greater than the change necessary in other cases, for commercial enamels require about 0.5 percent of cobalt oxide while as high as 3.5 percent of manganese oxide is required.

The individual members of the series were weighed up and mixed thoroughly by hand. They were then fritted in a gas-fired frit furnace, in crucibles holding a batch of about 4.5 pounds. In each case, the mixture was melted down, then kept in a molten state and stirred occasionally in order to eliminate the bubbles as much as possible. The melts were kept in this condition until a thread drawn between the fingers did not contain any lumps due to undissolved material nor any gas bubbles. When the fritting, which required about three hours, was completed, the enamel was poured into cold water in order to shatter the mass and thus facilitate the grinding. After being dried, the frit was weighed out with 8 percent of English ball clay and 1 percent of borax, the latter being added in a hot solution. The batches were ground in small porcelain ball mills with approximately 40 percent of water for about 20 hours, this time being required, in most cases, to grind the enamel just fine enough to pass a 150 mesh screen when washed through with water. The above procedure applies to all the enamels except Series F, in which part of the flint was added in the milling.

For trial pieces, 18 gauge Armco iron plates about 3 in. by 6 in. were used. These plates were scaled in an open fire kiln, after which they were pickled in a hydrochloric acid bath made up of 1 part commercial acid to 6 parts of water. After lying in the bath about 30 minutes they were scrubbed in clean water, then dipped in a very dilute solution of sodium carbonate to neutralize any acid remaining on the plates. They were then dried as quickly as possible in a steam coil drier. The enamels

were set up with a further addition of borax. The plates were sponged off and dipped, then set on racks and dried in the steam coil drier.

A small oil-fired muffle kiln, having a chamber 12 in. by 18 in. by 30 in. was used for the burning, the trials being placed on a grid large enough to hold three pieces. A thermo-couple was placed in the muffle with the junction as close as possible to the trials. The temperature and time required for properly burning each enamel were determined, if possible on the first trial in each set, and the rest of the trials were burned accordingly.

Tests. Three general tests were carried out on the trials, namely:

1. Microscopic examination of trials for bubbling,
2. Impact test for toughness or resistance to chipping,
3. Application and burning of white cover enamel.

The first test was made by examining the trials under a microscope and noting any tendency to bubble. The object of this test was to determine whether there was any relation between bonding power and formation of bubbles in a ground coat.

Series A: Increasing CoO
Improvement in quality, decreased pinholing.

Series B: Increase of NiO
Enamels not very smooth, decreased bubbling.

Series C: Increase of CoO, with decrease of NiO
No bubbling and good texture.

Series D: Increasing MnO
Some very small bubbles but enamel was very smooth.

Series E: Increase of CoO with decrease of MnO
Decrease in number of fine bubbles.

Series F: Increase of SiO_2
All pinholed, enamel not very smooth.

Series G: Increase of CaO
An increase in pinholing and bubbling

Series H: Decrease of B_2O_3
Pinholed and blistered, especially with low borax.

Series J: Increase of Sb_2O_3
Blistered and bubbled, decreasing to No. 3.

The second test was carried out in the apparatus shown in Fig. 1, which is similar to that used by R. Landrum[2] except for some few changes in construction, a 4 pound hammer with a hardened steel head being used instead of a 5 pound hammer. The plates were set on a piece of hardened steel, and the hammer allowed to drop from certain measured distances. The distance of fall was increased until the maximum height was reached at which chipping would not take place. Failure of the enamel was taken at the point where any chipping of the enamel occurred when scratched. It was found that any tendency to chip, which could not be noted otherwise, was brought out by a slight rubbing of the enamel. The distance of fall of the hammer was regulated to quarter inches, and the results obtained on the machine were fairly consistent when it is remembered that any variation in the thickness of coat, time of burning, etc., will change the quality of the enamel greatly.

The data for the impact tests is given below with the composition of the various enamels and their time of burning, etc. Table I in each series gives the molecular formula of the enamel while in Table II the percentage composition of the enamel and its temperature and time of burning are given. The temperature and time given are the means of the results on the ten trials in each particular set of series. Table III gives the data on the toughness test for the individual plates.

The results of the toughness tests are shown graphically in the curves, Fig. 2 and 3, an attempt being made to plot the curves so that the comparative effects of the various metallic oxides could be brought out more clearly.

From the curves, the following facts are obtained:—

(1) Cobalt and nickel oxides have about the same effect as regards toughness, while manganese oxide decreases the

[2] Test of Ten White Enamels, *Trans. Amer. Cer. Soc.*, Vol. XIV, Robt. Landrum.

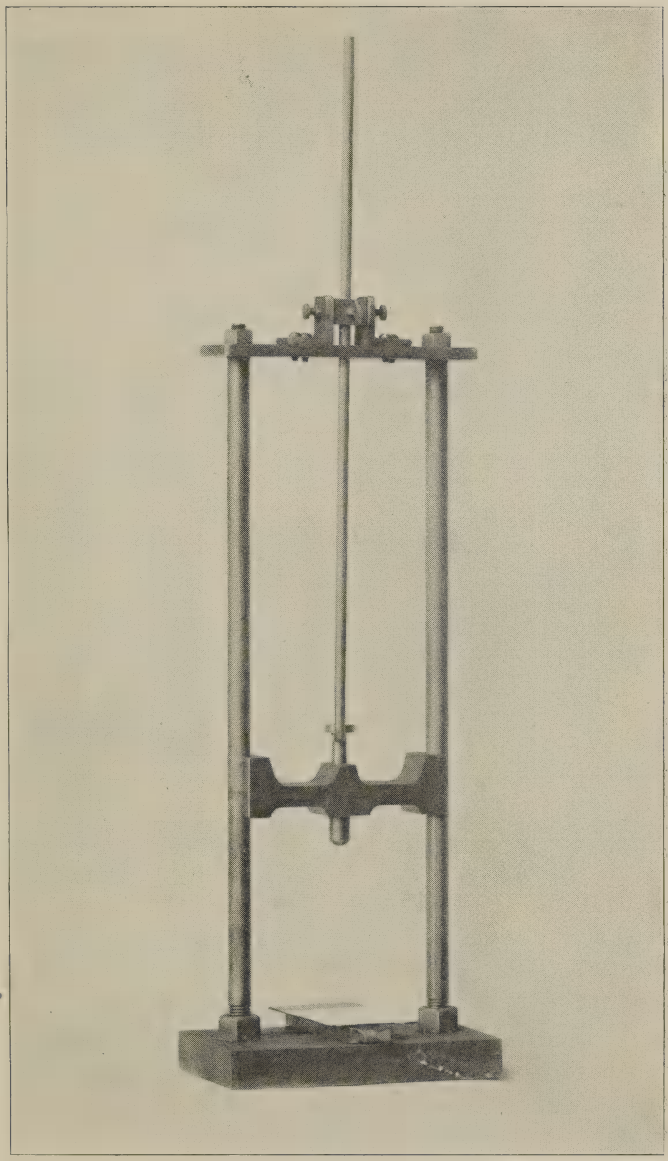
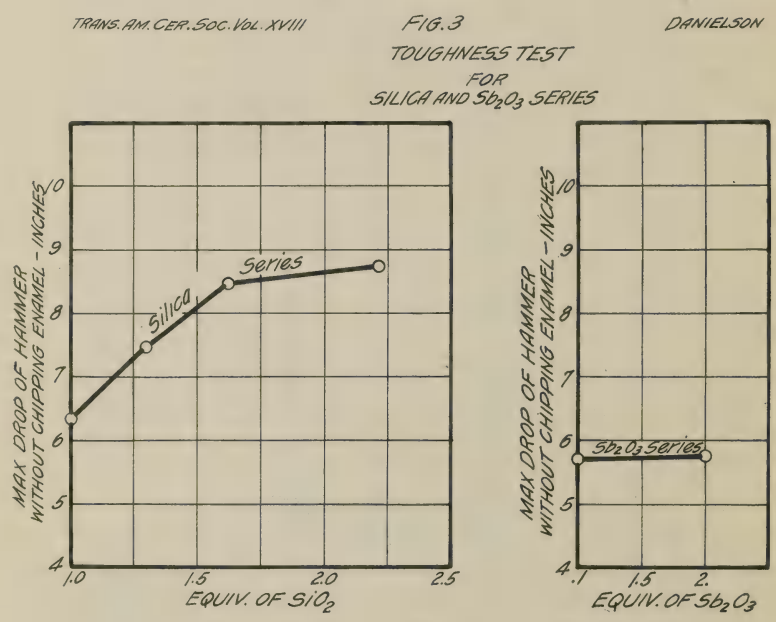


FIG. 1.

toughness very markedly. Additions of cobalt oxide to either manganese or nickel seems to increase the toughness. These results tend to show that cobalt oxide is an important constituent for developing toughness of ground coats. Furthermore, toughness is absolutely essential in any enamel which serves as a good ground.

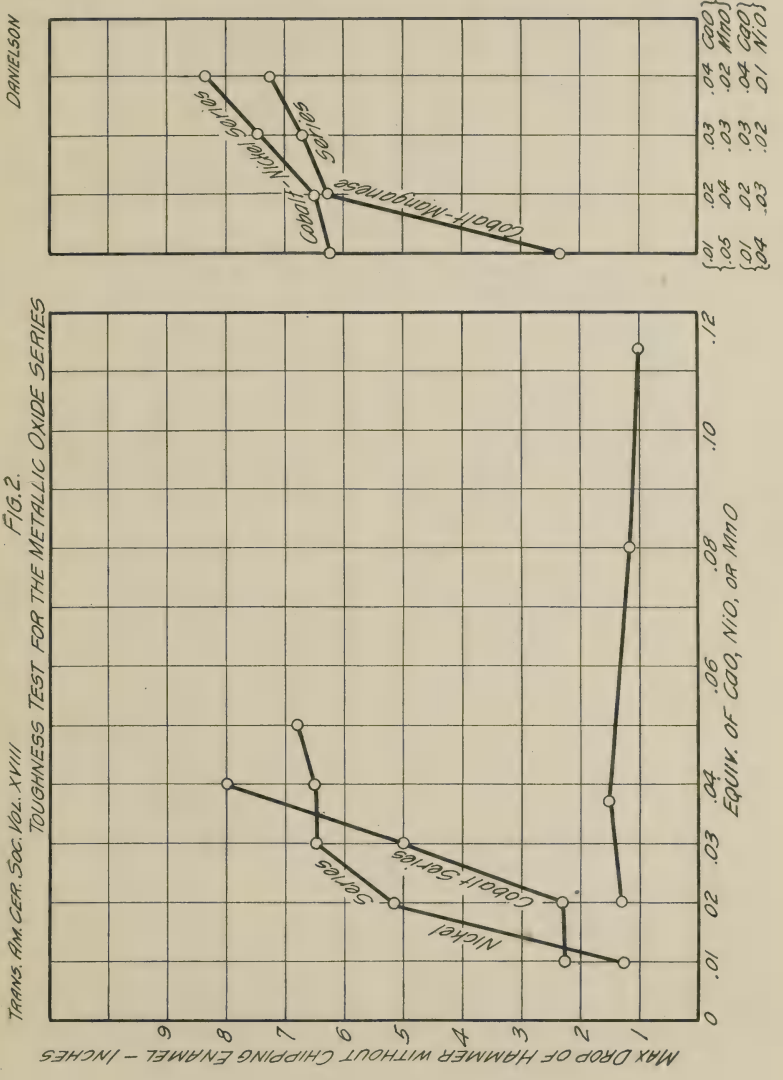
(2) The introduction of part of the flint into the enamel batch instead of into the frit itself seems to increase the tough-



ness, but at the same time makes the enamel more refractory. The enamels of this series have less the appearance of a glass which probably accounts for their greater toughness.

(3) Low borax or high lime enamels will not make good grounds as they are very porous and show very low toughness.

(4) Variations in the antimony oxide do not affect the toughness either way, but do add to the whiteness of the enamel. It might be possible that, by working in this direction, a white ground could be obtained.


DANIELSON
TRANS. AM. CER. SOC. VOL. XVIII
FIG. 2.
TOUGHNESS TEST FOR THE METALLIC OXIDE SERIES
MAX DROP OF HAMMER WITHOUT CHIPPING ENAMEL – INCHES
EQUIV. OF CoO, NiO, OR MnO
Nickel Series
Cobalt Series
Cobalt-Manganese
Cobalt
Nickel Series
Series
{.01 .05 .01 .04} {.02 .04 .02 .03} {.03 .03 .02 .02} {.04 .02 .04 .01} CoO MnO CoO NiO

THIRD TEST.

White Cover Enamel. Ability to take a finish white without bubbling. For this test a white cover coat of the following composition was used:

$$\left.\begin{matrix} .212\ K_2O \\ .632\ Na_2O \\ .156\ CaO \end{matrix}\right\} \left.\begin{matrix} .027\ Sb_2O_3 \\ .324\ B_2O_3 \\ .237\ Al_2O_3 \end{matrix}\right\} \left\{\begin{matrix} 2.19\ SiO_2 \\ .349\ F_2. \end{matrix}\right.$$

This has the following batch composition:

Borax	20
Feldspar	32
Flint	21.5
Soda	10
Saltpeter	2¼
Cryolite	16
Antimony	2½
Whiting	5

Additions in mill: { 7 percent tin oxide, ½ percent magnesia, 6 percent clay.

This enamel was sprayed on the ground coated plates, dried in a steam coil drier and then burned at 875°C in the same furnace that was used for burning the ground coats. This test is very important for if a ground cannot take a cover enamel without pinholing, blistering, or developing similar defects, it is of course worthless.

The results on this test are given below:

Series A.

A. 2 pinholed slightly,
A. 3 good enamel,
A. 4 excellent enamel.

Series B.

B. 1
B. 2
B. 3 } slightly bubbled.
B. 4
B. 5

Series C.

C. 1
C. 2 } all good enamels.
C. 3

Series D.

D. 1 good enamel,
D. 2 good enamel,
D. 3 excellent results,
D. 4 slightly bubbled.

Series E.

E. 1 slight pinholing,
E. 2 fair enamel,
E. 3 excellent enamel,
E. 4 excellent enamel.

Series F

F. Every member of series was pinholed.

Series G.

G. 1 bubbled,
G. 2 fair,
G. 3 fair,
G. 4 fair. } First coat was not smooth giving the finish enamels a wavy appearance.

Series H.

H. All pinholed and blistered.

Series J.

J. All blistered and bubbled but decreasing with increase of Sb_2O_3.

The results with the finish white show that best enamels were obtained on those first coats in which cobalt was present. In every series, the quality of the enamel was improved with the increase of cobalt. In no case were good results obtained with the plates coated with a white first coat, such defects as pinholing, blistering and bubbling being more or less present.

General Conclusions. In all this work, the results seem to be fairly consistent in that the different tests differentiate the poor enamels from the good enamels very well. There seems to be a very close connection between toughness, bubble formation and the ability to take a good finish enamel, except in the series in which flint was added to the enamel batch. This series gave very good results in the toughness test, but still did not make a good smooth ground coat, possibly due to the fact that adding the flint in this manner makes the enamel more refractory than it would be if the flint were added entirely in the frit. In order to make a good enamel, it is necessary that the ground coat soften sufficiently so that more fusible cover enamel can fuse into the ground. This point might be made use of in sheet iron enamel work by simply adding part of the flint to the frit for the ground coat thus producing a ground which would be tougher.

The best results have been obtained in this work by the use of cobalt and nickel or by a combination of the two. Fairly good results have also been obtained by the use of cobalt and manganese, but it is quite evident from the curves for cobalt, manganese, and cobalt + manganese that the toughness is due mainly to the cobalt. This is in accordance with the views of J. Grunwald[3], who says, "Every practical enameling expert knows that a ground enamel free from cobalt and nickel oxides is unobtainable. Although nickel oxide is excluded from white grounds where certainty is required on a large scale, it may replace cobalt oxide, wholly or partly, for many of the cheaper enamels. Many famous enameling firms, however, celebrated for excellent quality of their wares, only employed cobalt oxides for their grounds."

From the results of this work there appears to be a decided

[3] *Raw Materials of the Enamel Industry*, Julius Grunwald and H. H. Hodgson, p. 155.

advantage in the use of cobalt and nickel oxide, for all combinations of these oxides give good tough enamels. If cobalt oxide were used alone, a larger amount would have to be used in the batch to give the required toughness to the enamel. At the best this is only a small amount, and any error in the amount of cobalt oxide added would make considerable difference in the quality of the ground coat. In adding nickel oxide, about three parts are added for each part of cobalt oxide replaced. Nickel oxide is cheap as compared with cobalt oxide so there would be no increase in the cost of the enamel because of this addition of nickel oxide. Any slight error in the additions of nickel oxide or cobalt would not affect the ground very much as there would still be enough metallic oxides to make a good ground.

Another objection to the use of cobalt oxide alone is, that it develops a blue color on burning. On properly burning, a ground containing both cobalt and nickel oxides, the color gradually changes from a blue to a black. This is usually the only indication used in commercial work to denote the proper burning of a ground coat, and it is such a reliable indication that the use of cobalt and nickel is strongly advocated for the production of good coats.

From the results of this investigation, the following combination of the metallic oxides would probably be the best for a ground coat of high quality:

Combination No. 1 ..... { .4 percent CoO
.75 percent NiO

For a ground coat containing manganese oxide, the following combination would be the best, altho this would not produce a ground coat equal in quality to the one produced with combination as given above.

Combination No. 2 ..... { .5 percent CoO
1.5 percent MnO

Altho white ground coats may be produced by omitting the coloring oxides, they are not certain enough for commercial practice, and it is doubtful whether the savings derived from the use of a white ground coat would justify the risk taken in the production of high grade ware.

SERIES A — TABLE I. MOLECULAR COMPOSITION

NOS.	A-1	A-2	A-3	A-4
K_2O	.24	.23	.22	.21
Na_2O	.50	.50	.50	.50
CaO	.25	.25	.25	.25
CoO	.01	.02	.03	.04
Al_2O_3	.15	.15	.15	.15
B_2O_3	.50	.50	.50	.50
SiO_2	2.0	2.0	2.0	2.0
F_2	.25	.25	.25	.25

SERIES A — TABLE II. BATCH COMPOSITION IN PERCENT

NOS.	A-1	A-2	A-3	A-4
Potash-spar	27.0	27.1	27.2	27.3
Saltpeter	5.9	5.3	4.6	3.95
Fluorspar	6.3	6.3	6.35	6.4
Cobalt oxide	.26	.53	.80	1.07
Borax	30.6	30.8	31.0	31.2
Soda	8.3	8.6	8.6	8.65
Flint	21.2	21.3	21.4	21.5
Temperature of burn.		1000°	1000°	1000°
Time in seconds		100	65	70

SERIES A — TABLE III. TOUGHNESS TEST

NO. OF PIECES	GREATEST DROP BEFORE CHIPPING OCCURRED			
	SERIES A-1	SERIES A-2	SERIES A-3	SERIES A-4
1		2		
2	$2\frac{1}{4}$	$2\frac{1}{4}$	6	8
3	$2\frac{1}{4}$		5	$7\frac{1}{2}$
4		2	5	
5		$2\frac{1}{4}$	4	8
6		$2-\frac{3}{4}$	4	8
7		$2\frac{1}{4}$	5	6
8		$2\frac{1}{4}$		$8\frac{1}{4}$
9		2	5	8
Average	$2\frac{1}{4}$*	$2\frac{1}{4}$	5	8

* Series overburned.

SERIES B — TABLE I. EQUIVALENTS

	B-1	B-2	B-3	B-4	B-5
K_2O	.24	.23	.22	.21	.20
Na_2O	.50	.50	.50	.50	.50
CaO	.25	.25	.25	.25	.25
NiO	.01	.02	.03	.04	.05
Al_2O_3	.15	.15	.15	.15	.15
B_2O_3	.50	.50	.50	.50	.50
SiO_2	2.0	2.0	2.0	2.0	2.0
F_2	.25	.25	.25	.25	.25

SERIES B — TABLE II. BATCH COMPOSITION IN PERCENT

	B-1	B-2	B-3	B-4	B-5
Potash-spar	27.0	27.2	27.2	27.4	27.4
Saltpeter	5.85	5.25	5.26	5.29	5.3
Borax	30.8	31	31	31.2	31.3
Soda	8.55	8.6	8.6	8.63	8.7
Fluorspar	6.28	6.3	6.35	6.35	6.38
Ni_2O_3	.27	.588	.81	1.05	1.36
Flint	21.3	21.4	21.4	21.5	21.6
Temp. of burning.	1000°	990°	995°	985°	990°
Time in seconds...	70	75	100	100	100

SERIES B — TABLE III. TOUGHNESS TEST

NO. OF PIECE	GREATEST DROP BEFORE CHIPPING OCCURRED				
	SERIES B-1	SERIES B-2	SERIES B-3	SERIES B-4	SERIES B-5
1	1½	5½	6–¾	.....	6–¾
2	1¼	.....	6–¾	6–¾	.....
3	.....	.....	6–¾	7	.....
4	.....	5	.....	6½	7
5	1	.....	.....	6¾	6¾
6	1	5½	6½	6½	6–¾
7	1¼	5¼	6–¾	.....	6¼
8	1¼	7	.....	6½	7¼
9	.....	5¼	6¼	6¼	.....
10	1¼	.....	.....	.....	6¼
Average	1¼	5½	6–¾	6½	6–¾

SERIES C — TABLE I. EQUIVALENTS

	C-1	C-2	C-3	C-4
K_2O	.20	.2	.2	.2
Na_2O	.5	.5	.5	.5
CaO	.25	.25	.25	.25
CoO	.01	.02	.03	.04
NiO	.04	.03	.02	.01
Al_2O_3	.15	.15	.15	.15
B_2O_3	.5	.5	.5	.5
SiO_2	2.0	2.0	2.0	2.0
F_2	.25	.25	.25	.25

SERIES C — TABLE II. BATCH COMPOSITION IN PERCENT

	C-1	C-2	C-3	C-4
Potash-spar	27.4	27.4	27.4	27.4
Saltpeter	3.3	3.3	3.3	3.3
Borax	31	31	31	31
Soda	8.7	8.7	8.7	8.7
Fluorspar	6.4	6.4	6.4	6.4
Cobalt oxide	.28	.55	.82	1.09
Ni_2O_3	1.09	.82	.55	.28
Flint	21.6	21.6	21.6	21.6
Temp. of burning	985°	990°	965°	990°
Time in seconds	101	100	105	105

SERIES C — TABLE III. TOUGHNESS TEST

NO. OF PIECES	GREATEST DROP BEFORE CHIPPING OCCURRED			
	SERIES C-1	SERIES C-2	SERIES C-3	SERIES C-4
1	.....	6–¾	.....	.....
2	6–¾	6–¾	7¼	8–¾
3	6	6¼	7¼	8½
4	6	6	.....	8–¾
5	6¼	.....	7–¾	.....
6	6¼	6½	7½	.....
7	.....	6½	7–¾	.....
8	6¼	.....	.....	7
9	6¼	.....	7¼	8¼
10	6	6	.....	8
Average	6¼	6½	7½	8½

SERIES D — TABLE I. EQUIVALENTS

	D-1	D-2	D-3	D-4
K_2O	.24	.23	.21	.20
Na_2O	.50	.50	.50	.50
CaO	.24	.23	.21	.20
MnO	.02	.03	.07	.11
Al_2O_3	.15	.15	.15	.15
B_2O_3	.5	.5	.5	.5
SiO_2	2.0	2.0	2.0	2.0
F_2	.24	.23	.21	.20

SERIES D — TABLE II. BATCH COMPOSITION IN PERCENT

	D-1	D-2	D-3	D-4
Potash-spar	27.0	27.1	27.2	27.2
Saltpeter	5.9	5.3	4.07	3.30
Borax	30.8	30.9	31.0	30.8
Soda	8.5	8.55	8.6	8.6
Fluorspar	6.0	5.8	5.4	5.08
MnO_2	.57	1.07	2.15	3.25
Flint	21.3	21.4	21.5	21.5
Temp. of burning	950°	997°	995°	990°
Time in seconds	93	92	96	97

SERIES D — TABLE III. TOUGHNESS TEST

NO. OF PIECES	GREATEST DROP BEFORE CHIPPING OCCURRED			
	SERIES D-1	SERIES D-2	SERIES D-3	SERIES D-4
1	1½	2	1	.....
2	1¼	1½	1½	.....
3	.....	2	1	1
4	1¼	.....	1	.....
5	.....	1	.....	.....
6	.....	.....	.....	.....
7	.....	.....	.....	1
8	1¼	1	1	.....
9	1	.....	.....	1
10	.....	.....	.....	1
Average	1¼	1½	1	1

SERIES E — TABLE I. EQUIVALENTS

	E-1	E-2	E-3	E-4
K_2O	.23	.23	.23	.23
Na_2O	.48	.48	.48	.48
CaO	.23	.23	.23	.23
CoO	.01	.02	.03	.04
MnO	.05	.04	.03	.02
Al_2O_3	.15	.15	.15	.15
B_2O_3	.50	.50	.50	.50
SiO_2	2.0	2.0	2.0	2.0
F_2	.23	.23	.23	.23

SERIES E — TABLE II. BATCH COMPOSITION IN PERCENT

	E-1	E-2	E-3	E-4
Potash-spar	27.1	27.1	27.1	27.1
Saltpeter	5.24	5.24	5.24	5.24
Borax	31.0	31.0	31.0	31.0
Soda	7.9	7.9	7.9	7.9
Fluorspar	5.8	5.8	5.8	5.8
Cobalt oxide	.27	.54	.81	1.08
MnO_2	1.41	1.13	.85	.56
Flint	21.4	21.4	21.4	21.4
Temp. of burning	995°	975°	950°	985°
Time in seconds	119	97	87	98

SERIES E — TABLE III. TOUGHNESS TEST

NO. OF PIECES	GREATEST DROP BEFORE CHIPPING OCCURRED			
	SERIES E-1	SERIES E-2	SERIES E-3	SERIES E-4
1	3	6–¾	6–¾	7
2	.....	7	.....	7
3	.....	6¼	6–¾	7¼
4	1½	.....	.....	7½
5	3	.....	.....	7
6	1½	6	6½	.....
7	2–¾	.....	.....	7–¾
8	.....	6¼	7¼	7¼
9	3	.....	7	.....
10	2–¾	6¼	6–¾	.....
Average	2–¾	6½	6–¾	7¼

SERIES F — TABLE I. EQUIVALENTS

	F-1	F-2	F-3	F-4	F-5
K_2O	.25	.25	.25	.25	.25
Na_2O	.50	.50	.50	.50	.50
CaO	.25	.25	.25	.25	.25
Al_2O_3	.15	.15	.15	.15	.15
B_2O_3	.50	.50	.50	.50	.50
SiO_2	1.0	1.3	1.6	1.9	2.2
F_2	.25	.25	.25	.25	.25
Sb_2O_3	.05	.05	.05	.05	.05

SERIES F — TABLE II. BATCH COMPOSITION

F-1		F-2		F-4	
K-Spar	83.70	Frit	187.5	Frit	187.5
Saltpeter	20.20	Flint	18	Flint	54
Borax	95.90				
Soda	26.50	F-3		F-5	
Fluorspar	19.50				
Sb_2O_3	14.40	Frit	187.5	Frit	187.5
Flint	6.00	Flint	36	Flint	72

	TEMP. OF BURNING	TIME, SEC.
F-1	960°	80
F-2	940°	94
F-3	965°	80
F-4	975°	105
F-5	997°	83

SERIES F — TABLE III. TOUGHNESS TEST

NO. OF PIECE	GREATEST DROP BEFORE CHIPPING OCCURRED				
	SERIES F-1	SERIES F-2	SERIES F-3	SERIES F-4	SERIES F-5
1	.....	.....	9	.....	9
2	.....	.....	.....	.....	$8\frac{1}{4}$
3	$6-\frac{3}{4}$	.....	$8-\frac{3}{4}$	.....	.....
4	.....	8	$8-\frac{3}{4}$	.....	9
5	.....	.....	$8\frac{1}{4}$	.....	9
6	.....	.....	.....	.....	9
7	$6\frac{1}{4}$	8	$8\frac{1}{2}$	.....	9
8	$6-\frac{3}{4}$	$7\frac{1}{2}$	8	.....	$8-\frac{3}{4}$
9	$6\frac{1}{4}$	$7-\frac{3}{4}$	.....	.....	.....
10	.....	8	.....	.....	.....
Average	$6\frac{1}{2}$	$7-\frac{3}{4}$	$8\frac{1}{2}$	.....	9

SERIES G — TABLE I. EQUIVALENTS

	G-1	G-2	G-3	G-4	G-5
K_2O	.20	.15	.15	.15	.15
Na_2O	.50	.50	.45	.40	.35
CaO	.30	.35	.40	.45	.50
Al_2O_3	.15	.15	.15	.15	.15
B_2O_3	.5	.5	.5	.5	.5
Sb_2O_3	.05	.05	.05	.05	.05
SiO_2	2.0	2.0	2.0	2.0	2.0
F_2	.3	.35	.40	.45	.50

SERIES G — TABLE II. BATCH COMPOSITION IN PERCENT

	G-1	G-2	G-3	G-4	G-5
Potash-spar	26.2	26.6	26.8	26.9	27.0
Saltpeter	3.16	.....	.....	.....	.....
Borax	30.0	30.5	30.7	30.8	30.9
Soda ash	8.30	8.45	6.8	5.13	3.44
Fluorspar	7.3	8.73	10.0	11.3	12.6
Sb_2O_3	4.50	4.58	4.59	4.60	4.62
SiO_2	20.7	21.0	21.2	21.3	21.4
Temp. of burning	997°	999°	1000°	975°	950°
Time in seconds	123	75	76	118	130

SERIES G — TABLE III. TOUGHNESS

G–1 and G–2 — bubbled.
G–3, G–4 and G–5 — smooth.
Below $1\frac{1}{2}$ all failed.

SERIES H — TABLE I. EQUIVALENTS

	H-1	H-2	H-3
K_2O	.25	.25	.25
Na_2O	.50	.50	.50
CaO	.25	.25	.25
Al_2O_3	.15	.15	.15
B_2O_3	.4	.3	.2
SiO_2	2.0	2.0	2.0
F_2	.25	.25	.25
Sb_2O_3	.05	.05	.05

SERIES H — TABLE II. BATCH COMPOSITION IN PERCENT

	H-1	H-2	H-3
Potash-spar	26.8	28.0	29.4
Saltpeter	6.5	6.8	7.1
Borax	24.5	19.2	13.4
Soda	10.2	12.4	14.9
Fluorspar	6.35	6.54	6.85
Sb_2O_3	4.6	4.83	5.06
Flint	21.2	22.1	23.2
Temp. of burning	975°	985°	993°
Time in seconds	110	106	110

SERIES H — TABLE III. TOUGHNESS

H-1, H-2 and H-3 all failed under 2 inches, and all bubbled.

SERIES J — TABLE I. EQUIVALENTS

	J-1	J-2	J-3
K_2O	.25	.25	.25
Na_2O	.50	.50	.50
CaO	.25	.25	.25
Al_2O_3	.15	.15	.15
B_2O_3	.5	.5	.5
Sb_2O_3	.10	.15	.20
SiO_2	2.0	2.0	2.0
F_2	.25	.25	.25

SERIES J — TABLE II. BATCH COMPOSITION IN PERCENT

	J-1	J-2	J-3
Potash-spar	24.6	23.2	22.7
Saltpeter	5.93	5.60	5.48
Borax	28.1	26.5	25.9
Soda	7.8	7.36	7.2
Fluorspar	5.72	5.42	5.30
Sb_2O_3	8.46	13.4	15.6
Flint	19.4	18.2	17.9
Temp. of burning	975°	976°	970°
Time in seconds	77	72	69

SERIES J — TABLE III. TOUGHNESS

NO. OF PIECE	GREATEST DROP BEFORE CHIPPING OCCURRED		
	J-1	J-2	J-3
1	.....	.....	.....
2	.....	.....	.....
3	5–½	.....	.....
4	5–½	.....	5–¾
5	5	.....	.....
6	5–¾	.....	5–¾
7	6	.....	.....
8	6	.....	6
9	6	.....	5–½
10	.....	.....	.....
Average	5–¾	.....	5–¾

ON THE ATTAINMENT OF RELIABLE TEMPERATURE MEASUREMENTS IN THE CERAMIC INDUSTRIES BY MEANS OF THEMOCOUPLES[1]

BY WALTER A. HULL

Burned clay products of all sorts can be burned without the aid of pyrometers. It is not absolutely necessary that kiln temperatures should be recorded, or even known, at any stage in the burn. On the other hand, there are probably not many cases where fuel and labor could not be saved and the time of burning reduced, by the use of burning schedules worked out in the light of time-temperature burning records. Such practice makes for efficiency in burning and also tends to reduce the uncertainty of getting consistently good burns. Opposed to these advantages there are the following difficulties to reckon with:

(a) The initial cost of pyrometer equipment.
(b) Opposition on the part of kiln burners.
(c) The unreliability of temperature measurements made with thermo-electric pyrometers.

The pyrometer equipment should be regarded as a legitimate and permanent investment. Whether the investment is justified, undoubtedly depends, in many cases, on the validity of the other two disadvantages.

Opposition on the part of kiln burners may be a real factor to reckon with or it may not. The kiln burner may have suspicions and misgivings that are both genuine and formidable, but which would disappear if he understood and believed that the purpose of the innovation was to be an aid to the burning the same as better kilns or better fuel. It would also contribute to his peace of mind if he were sure that the new apparatus would not call for unreasonable educational qualifications on his part.

[1] By permission of the Director, Bureau of Standards.

The writer wishes to make two suggestions in this connection:

1. Don't install any part of the temperature measuring equipment in "The Office."

2. Don't revolutionize your burning—at any rate not right away.

In the first place, not many men are competent to supervise the burning without getting out into the plant where the burning is being done. The installation of a galvanometer or potentiometer for measuring kiln temperatures in the office is apt to mean that someone who stays in the office most of the time means to keep tab on the burning and on the burners by means of that instrument alone. To the burner, this does not mean supervision, but interference. In most cases, this view would be correct. The instrument should be near the kilns, where the burners can watch it and where the superintendent can consult it and consult with his burners at the same time.

As for revolutionizing the burning, that should not be the purpose of temperature measurement. The pyrometer should be considered rather as an aid to studying kiln problems and gradually standardizing kiln practice. Let the burner get used to having the pyrometer on the job; leave him alone with it to get acquainted with it and accept it as a part of the equipment. If he is anywhere near the right sort of a burner, he will gradually get to depending on it and, having voluntarily turned to the pyrometer for help, he will be glad to give his assistance in making a systematic study of the information it supplies.

As to the third, and perhaps the most serious of all objections to the pyrometer, the unreliability of its temperature measurements, there is no question that a number of pyrometric installations have proven to be worse than useless because they have failed, utterly, to measure temperatures correctly. If such results were to be expected and there were no way of effectively guarding against them, it would be better to dismiss the subject of pyrometers from practical consideration. Fortunately, such is not the case. Dependable pyrometers can be secured and with reasonable care, they will remain dependable. Furthermore, their accuracy can be tested, definitely, by positive methods, at

any time, so that there should be no uncertainty as to their accuracy. It is the purpose of the following paragraphs to put on record information of a purely practical nature which may be of value in the purchase and use of pyrometric equipment.

The type of pyrometer under consideration consists essentially of a thermocouple, connected to a galvanometer or a potentiometer. The thermocouple consists of two pieces of wire, of different composition, welded together. The weld joining the two wires is called the junction. In ordinary practice the thermocouple is placed, permanently, in a protection tube with one closed end. Protection tubes are commonly made of impervious glazed porcelain which protects the thermocouple from the action of the kiln gases. Within the protection tube is a small tube which keeps the two wires of the thermocouple separate except at the junction.

Protection tubes of material other than porcelain and adapted to special conditions are on the market. For use in kilns, generally, porcelain tubes are to be recommended. In cases where the temperatures do not exceed 1100°C, protection tubes of fused silica can be used satisfactorily.

Thermocouples suitable for use in kilns may be classed as:

(a) Platinum couples,
(b) Base metal couples.

Platinum couples are relatively expensive but they have the advantage of being adapted to high as well as low temperatures. With suitable care they can be used, at all kiln temperatures, for years without serious deterioration.

Base metal couples are comparatively inexpensive and may be accurate and reliable. They are not, however, capable of withstanding, for long periods, the high temperatures attained in many ceramic kilns. For low temperatures base metal couples are worthy of consideration. Several kinds are available of which the best known is the copper-constantan element. The copper-constantan couple, made of suitable wire, is capable of giving accurate results at all temperatures up to 500° and perhaps

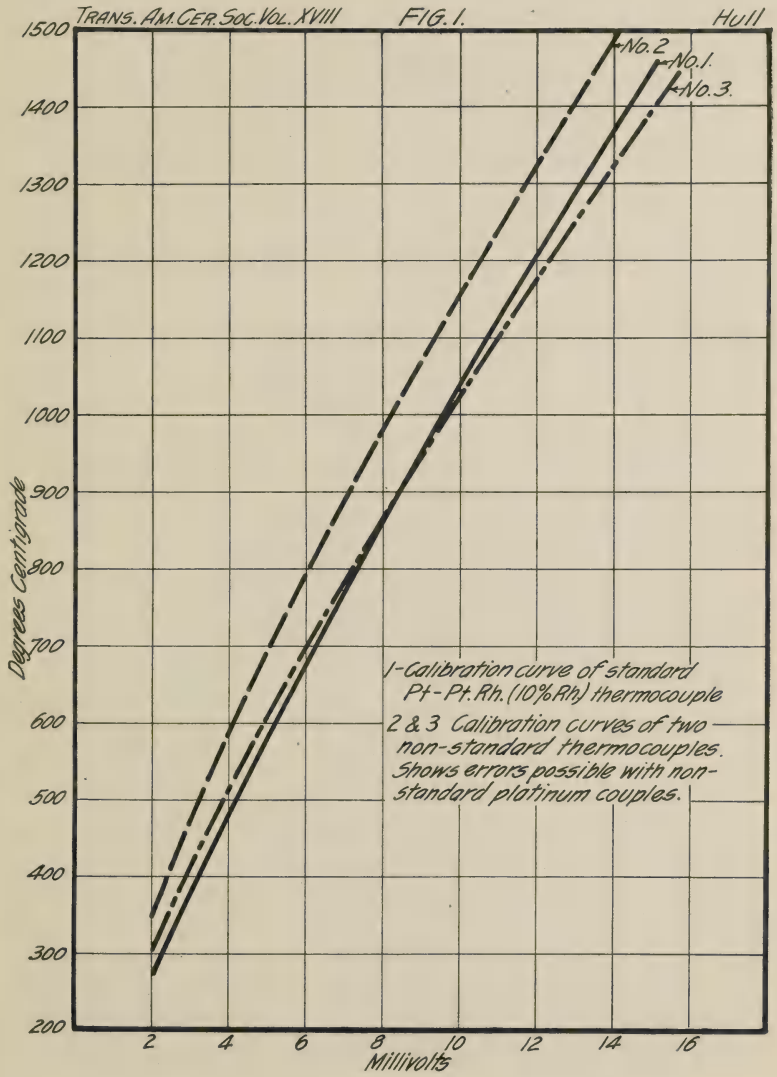
somewhat higher.[2] Deterioration, due to oxidation, would probably take place rather rapidly in copper-constantan couples in ceramic kilns at temperatures higher than 500°C. Iron-constantan couples are used in many industrial operations at temperatures up to 1000°C (1800°F.) at which temperatures they have, when suitably protected, a reasonable life such that the renewal costs are relatively unimportant. Other base metal couples now on the market are said to give satisfactory service up to 1100°C. (about 2000°F.); the prospective user should have satisfactory evidence of the suitability of the apparatus before adopting pyrometric equipment including them.

When a thermocouple is in place, with the junction of the two dissimilar wires heated, the electromotive force set up is measured with a galvanometer or potentiometer. For a given thermocouple the amount or magnitude of the electromotive force set up at any time depends on the temperature of the junction. This assumes that the cold junctions, which are the contacts between the outer ends of the wires of the thermocouple and the copper wires leading to the measuring instrument, are kept at a constant temperature. Since a given thermocouple, at a given temperature, always sets up the same electromotive force, the temperatures corresponding to various values of electromotive force for a given couple, when determined, can be shown on the scale of the instrument to be used with that couple, so that, when the deflection of the needle indicates an electromotive force of say five millivolts, it also indicates a temperature of say 575°C. It is customary to have, in galvanometers for use with thermocouples, two scales, parallel to each other, one in millivolts and the other in degrees. The temperature scale holds good only for thermocouples that are alike. If a galvanometer has a temperature scale that is correct for a standard platinum, platinum-rhodium couple, of which one wire is pure platinum and the other wire is 90 percent platinum and 10 percent rhodium, this galvanometer with this scale can be used with any

[2] The Thermo Element as a Precision Thermometer, by Walter P. White. *The Physical Review*, Vol. XXXI, No. 2, August, 1910.

The Calibration of Copper-Constantan Thermoelements. By W. P. White, H. C. Dickinson and E. F. Mueller. *The Physical Review*, Vol. XXXI, No. 2, August 1910.

Measurement of High Temperatures. By Burgess and Le Chatelier, page 167.


TRANS. AM. CER. SOC. VOL. XVIII
FIG. 1.
HULL
No. 2
No. 1.
No. 3.
1-Calibration curve of standard Pt-Pt. Rh. (10% Rh) thermocouple
2 & 3 Calibration curves of two non-standard thermocouples. Shows errors possible with non-standard platinum couples.
Degrees Centigrade
Millivolts
1500
1400
1300
1200
1100
1000
900
800
700
600
500
400
300
200
2
4
6
8
10
12
14
16

standard platinum, platinum-rhodium couple; but if it were used with a platinum couple which was not standard, because of some deviation from the proper composition of one or both of the wires, the temperature readings would not be correct. This is due, of course, to the fact that at a given temperature the non-standard couple does not set up the same electromotive force as the standard couple.

In the clay working industry it is desirable, ordinarily, to have a single galvanometer or potentiometer and to be able to read temperatures from a number of thermocouples with this one instrument. By the use of a suitable arrangement of leads and a selective switch, this is perfectly feasible and convenient, provided the temperature scale of the instrument holds good for all the thermocouples. This raises the question of whether a manufacturer can obtain any number of thermocouples which are alike in their temperature readings and whether a couple purchased this year may be expected to check up with others, which have been in use for several years. This is a question which justifies somewhat thorough treatment.

There are platinum couples on the market which do not check up with standard platinum couples. Furthermore, such couples may not check up with each other. As an illustration, the calibration curves of three platinum couples is shown in Fig. 1. Curve No. 1 shows the calibration of a normal, or standard, platinum platinum-rhodium couple. Curves No. 2 and No. 3 show the calibration of two other platinum platinum-rhodium couples. These were two out of a lot of several couples that were purchased together. It is evident that if a manufacturer were to attempt to use the last two couples without taking into account the individual calibration of each one, the results would be misleading and worse than useless. However, it is not necessary to accept such thermocouples. It is a simple matter to specify standard, or normal, platinum platinum-rhodium couples of standard calibration and to find out whether the couples furnished check up with that calibration. The calibra-

tion[3] for couples of pure platinum and 90 percent platinum, 10 percent rhodium is as follows:

TABLE I

Standard calibration for Pt. Pt.-Rh. (10 percent Rh.) thermo-element, cold junction at O° C.

MILLIVOLTS	DEGREES CENTIGRADE	MILLIVOLTS	DEGREES CENTIGRADE
0	00	10	1037.3
1	147.1	11	1122.2
2	265.4	12	1205.9
3	374.3	13	1289.3
4	478.1	14	1372.4
5	578.3	15	1454.8
6	675.3	16	1537.5
7	769.5	17	1620.9
8	861.1	18	1704.3
9	950.4	..	.....

The manufacturer or dealer supplying thermocouples should furnish a certificate giving the calibration of each couple. If at any time the calibration of a thermocouple is in doubt, it can be sent to the Bureau of Standards at Washington and its calibration determined. Or, if suitable equipment is available, couples of doubtful calibration can be checked up by comparing them with a couple of known calibration. A further discussion of this method will be given further on.

Some evidence as to the reproducibility of thermocouples, that is, of the possibility of manufacturing thermocouples which are interchangeable, is given in the following[4] "——we may cite the case of the well-known 10 percent platinum-rhodium normal thermocouples of Heraeus. They are reported to have maintained the following constancy for the past six years:"

[3] L. H. Adams. Calibration Tables for Copper-Constantan and Platinum—Platinum-Rhodium Thermoelements. *The Journal of the American Chemical Society*, Vol. XXXVI, No. 1, January 1914.

[4] *Circular: Bureau Standards, No. 7.* Pyrometer Testing and Heat Measurements.

[5] Burgess and Le Chantelier. *Measurement of High Temperature*, p. 164.

Average E. M. F. at 1000° C.

Year	Millivolts
1904	9.52
1905	9.53
1906	9.53
1907	9.57
1908	9.59
1909	9.55

It may be said in this connection that the Heraeus couples are still holding up to this creditable showing. American manufacturers have not, so far, had this degree of success in the manufacture of platinum couples. However, the matter of most importance to the user is that couples of standard calibration be obtained, which, as already stated, can be accomplished by specifying standard couples and comparing the calibration of each one with the standard calibration. The importance of this can hardly be exaggerated.

The Galvanometer or Potentiometer. Reliable pyrometer galvanometers and potentiometers have been produced by a number of American and European firms. Such an instrument may be expected to give satisfactory service for a number of years, and it is best to purchase a high class instrument at the outset. Because of the relatively long life of these instruments the first cost may properly be regarded as a secondary consideration in the selection of this part of the equipment.

Several types of pyrometer instruments are available. The most valuable sort of instrument for use in connection with kiln burning is the multiple recorder type. The advantage of multiple recorders is that they automatically make record of the temperatures indicated by a number of couples. Such records may be made of great value in the attainment of efficiency in burning. Indicating instruments give as reliable readings as recording ones and by the use of a suitable commutator or switch arrangement, one indicating instrument will serve to take the readings from any number of thermocouples. The difficulty is that in order to make satisfactory records, some responsible per-

son must be at the instrument at certain times, to take the readings and write them down. When there are not enough responsible persons available for what has to be done, the recording may be neglected and finally discontinued.

Portable Instruments. — It may be desirable in certain conditions to take temperature readings in parts of a kiln where no thermocouples are regularly located. Small portable galvanometers, of the pivoted type are convenient for this purpose. While not as sensitive as the less robust stationary form, these instruments, if of good make, are reasonably accurate. They are compact and do not require careful leveling. Special thermocouple protection tubes are now on the market which resist sudden heating and cooling, so that thermocouples may safely be inserted in and withdrawn from regions of high temperature. Such equipment, while convenient for certain purposes, should be regarded as secondary or auxiliary to the permanent installation.

Installation of Equipment. The pyrometer should be regarded as an important, permanent and substantial part of plant equipment. High class apparatus should be secured and then it should be treated as high class apparatus. Both requirements must be met in order to attain reliable temperature measurements by means of thermocouples. The installation may properly be taken up under three headings:

Placing the Galvanometer or Potentiometer. Perhaps the first consideration in the location of the galvanometer or potentiometer is convenience for the kiln burner. The experienced kiln burner already has a sixth sense; the pyrometer is meant to provide him with a seventh, and to fulfill its purpose it must be conveniently and comfortably accessible to him. It should be in a permanent building, reasonably clean, reasonably free from jars and vibrations. Good light should be provided and-perhaps some means of heating. The instrument itself should be placed on something solid where it will remain level. It is well, in a clay ware plant, to provide an outer cabinet with a glass front to assist in keeping dust out of the instrument.

Installation of Thermocouples. It is not necessary and perhaps in most cases not desirable, that thermocouples should

be permanently installed in kilns. Each thermocouple should be permanently placed in its protection tube, the two wires of course being kept insulated from each other. A suitable fixture, fitted and fastened to the open end of the protection tube, provides fastenings for the ends of the wires of the couple and binding posts for the wires that connect it with the galvanometer. This makes a simple, self-contained unit, which, when cold, can safely be taken from its position in one kiln and placed in another.

There is a variety of opinions as to the best location for thermocouples in the kiln. Frequently they are placed in small openings through the kiln wall. In each of these openings should be permanently installed, a strong, reasonably thick, fire clay tube, closed at the inner end. The end of the fire clay tube should project in several inches beyond the inner surface of the kiln wall. In this way a suitable position may be provided for the thermocouple without serious danger of accident to the porcelain protection tube.

Wiring. Connecting lines from thermocouples to galvanometer should be of insulated copper wire. The wire should not be excessively fine. The lines should be permanently installed by a competent man. All permanent connections should be soldered; otherwise they are likely to become corroded and give rise to serious trouble. Temporary connections should be made by means of suitable copper connectors and not by twisting the wires. Switches, of copper, should be kept bright at contacts. Switches of the knife or plug type are to be recommended. The object of these precautions is, of course, to avoid excessive resistance in the circuit, as all resistances affect the readings. The resistance of the copper wires themselves, unless they are extremely long or excessively small, is negligible, but the importance of good connections, where connections are necessary at all, can hardly be over-emphasized.

Care of Platinum Thermocouples. The one essential requirement in the use of platinum therocouples in ceramic kilns is that the couple itself shall be protected, by a suitable tube, from contamination. Platinum has a tendency to absorb impurities, especially metallic vapors, even at relatively low temperatures. If subject to reducing conditions in the presence of sili-

cates platinum is affected even at low red heats. Contamination can be entirely avoided by always using the couple in an impervious, closed end tube. As stated before, porcelain protection tubes are best for high temperatures. If the kiln temperatures do not exceed 1000° or 1100° C. protection tubes of fused silica serve the purpose as well as porcelain. Fused silica tubes have the advantage of resisting sudden heating and cooling better than porcelain, but ordinarily there is no occasion to subject the tube to sudden heating or cooling. Fused silica tubes do not last well in high temperature service.

After long service, a platinum couple may finally deteriorate, due to crystallization. In case of either contamination or crystallization the couple may be restored, partially, at least, by annealing. This is commonly done by suspending the couple in air and heating it by passing an electric current through it. The wire should be heated to a high temperature, and this temperature maintained for an hour or more. The temperature of annealing should be a reasonably close approach to the maximum temperature that the couple will stand without some spot in the wire getting too hot and parting. In case this should occur, the couple can be mended by welding.

Welding Platinum Couples. One of the manifestations of deterioration of a couple by either contamination or crystallization, is brittleness and mechanical weakness. The proper treatment is annealing followed by mending by welding. The process of welding consists of butting the two ends together in a hot flame, such as the oxyhydrogen or the oxygen-illuminating gas flame or an electric arc. If the electric arc is to be used, cored carbons are to be strongly recommended. With ordinary carbons the arc has too much tendency to wander. In any case, colored glasses should be used for the protection of the eyes. In welding in the flame of the electric arc, it is best to have the carbons so placed in relation to the eye that the line of vision is over the brightest part of the arc rather than through it. This makes it possible to see the ends of the wires of the couple much more clearly. These ends should be gradually moved in toward the brightest part of the arc until fusion begins, and then joined together.

Calibration of Thermocouples. If there is a question as to the correctness of the readings of any particular thermocouple, it is best to have it calibrated. Couples which need correction should be sent to reputable physical laboratories of which there are several in the country. Among others, the Bureau of Standards[6] was established by the government for the purpose of furnishing just such service to the public. If several thermo-couples are in use, or are to be in use, it may pay to provide means of checking them up at the plant. This can be done with a sufficient degree of accuracy by comparing the couple in question with one of known calibration. The couple that is to be used as the standard of comparison should not be used except for this purpose. In comparing a given thermocouple with a standard couple, all cold junctions should be kept at the same temperature and the two hot junctions should be so placed that the temperature of the two will always be alike. This is conveniently accomplished by placing both couples in a piece of small, four-hole clay tubing with their junctions close together at one end of this tube and heating them in a small electric tube furnace. A suitable tube furnace can be purchased or one can be made by winding a strip of platinum foil or a piece of fine platinum wire on a piece of porcelain protection tube. The coil may be held in place by covering with a paste of kaolin or porcelain body and baking it on. Then a further covering of some good heat insulating material is put on. The tube furnace is heated by passing an electric current through the coil. The current should be under control. A water rheostat will answer for this, so that any desired temperature may be maintained, nearly constant. To establish a point on the calibration curve of the unknown couple, hold the temperature of the furnace constant, or nearly so, and take a millivolt reading from each couple. The actual temperature of the furnace is readily determined from the reading of the known couple and the e. m. f. or millivoltage of the unknown corresponding to this actual temperature is shown by the millivolt reading of the unknown. It is well to assume that the furnace

[6] *Circular of the Bureau of Standards, No. 7,* Pyrometer Testing and Heat Measurements, p. 17.

temperature is not exactly constant while the readings are being taken and to proceed as follows:

1. Read the known couple.
2. Read the unknown couple.
3. Read the known couple again.
4. Use the average of the two readings of the known couple for comparison with the reading of the unknown. Any number of points in the calibration curve of the unknown couple may be located by this method and the curve drawn in through these points.

In checking up a couple by this method, two precautions must be taken:

1. Be sure the couple junctions are so arranged as to be at the same temperature. To secure this condition, the two junctions should be of about the same size and be located close together near the center of the tube and on a level with each other, not one above the other.

2. Watch for errors due to induction effect from the coil. Such errors are not likely to be found if couples are placed as outlined above. If present they can be detected by reversing the current through the coil.

The galvanometer or potentiometer should not get out of order nor its calibration require checking up until it has been in service for a long time. When it does need attention, it may be sent to a laboratory that is prepared to adjust and repair instruments for electrical measurements.

The present highly developed and highly practical forms of the thermoelectric pyrometer make this aid to the attainment of better efficiency and better results in kiln burning, well worthy of consideration. The first cost, the possible opposition of kiln burners and the danger of not attaining consistently reliable temperature measurements must be taken into consideration. However, a careful study of the last two of these contingencies should satisfactorily dispose of them, in which case the first cost of the installation would look only moderately formidable when viewed

in comparison with the values that are at stake in the burning of ceramic products. Study and supervision should be given to the selection, installation and use of pyrometric apparatus, but if the same intelligence and perseverance that are necessary in other departments of the factory practice are devoted to attaining the good service and full benefits of the pyrometric equipment, it may be depended upon to give a good account of itself, in contributing to the success of the plant.

THE CONSTITUTION AND MICROSTRUCTURE OF PORCELAIN[1]

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INTRODUCTION

The question of the microstructure and constitution of bodies consisting of mixtures of feldspar, quartz and clay substance is indeed an interesting one both scientifically and technically. Feldspar, quartz and clay are the raw materials which enter into the manufacture of whiteware (porous, non-vitreous) bodies as well as in the manufacture of porcelain and allied (dense, vitreous) ware. Of interest scientifically are the physical and chemical changes affecting these materials which take place at temperatures in the vicinity of those employed in the burning of commercial ware. These can be studied by the microscopical examination of the burned product. Furthermore, of great technical importance would be the ability to determine by means of microscopic methods the approximate composition of a body and above all the temperature range at which the body was burned. Within limits, all this data can be determined by petrographic microscopical means. Apparently, therefore, this method of investigation gives much information that cannot be determined by purely chemical means and ought to become an important tool in the hands of the ceramic investigator as well as the technical ceramist.

LITERATURE

The literature on this subject is voluminous. The first investigators of the constitution of porcelain were Brongiart and Malaguti[2]. They observed that a mixture of chemically pure silica, alumina, lime (as carbonate) and potash (as carbonate) in the proportions by weight which were obtained by the analysis of Sèvres Porcelain gave a more fusible mass than when the

[1] By permission of the Director, Bureau of Standards.
[2] *Pogg. Ann.* (1843) 89.

oxides were originally in chemical combination with each other. Von Bischof[3] showed that the chemical constitution of the raw materials was of importance in the formation of porcelain. According to G. Rose[4] porcelain is either a mixture of feldspar, glass and kaolin, or what he considered more probable, the feldspar and kaolin are, either wholly or partially, in chemical combination. According to the microscopic investigation of Behrens[5], the kaolin is more soluble in the molten feldspar than the quartz in the formation of porcelain. The quartz partly retains its original crystalline condition while the kaolin exists entirely in the melt. When porcelains containing quartz are burned most of the quartz splinters become rounded through solution and are covered with a silicate zone which protects the quartz from further solution, especially since the groundmass is not sufficiently fluid for a diffusion of the different silicates. According to Behrens, the groundmass of a porcelain undergoes devitrification. The material separating from the glass is dissolved more slowly in hydrofluoric acid than the quartz and glass. Seger[6] reaches the following conclusions concerning his investigations on the changes involved in the burning of porcelain. "One or more constituents melt and have a solvent effect on the rest of the constituents. The undissolved portion forms a solid skeleton which holds together the form of the mass. At higher temperatures solution proceeds, and the pores of the mass are more or less filled with the liquid phase, until finally the pores are completely filled and closed." Seger deduced also that the influence of the feldspar on the kaolin is less than on the quartz.

Of interest are the microscopic studies of Hussak[7] who examined thin sections of several porcelains burned at different temperatures. According to him a thin section of a very low burned specimen showed no change in the mixture, all the constituents being doubly refracting and clearly distingushable from each other. With an increase in burning temperature, the feldspar melts and the formation of glass begins; the quartz is sharp grained, the amount of kaolin is, however, apparently smaller.

[3] *Notizbl.* (1875) 162.
[4] *Pogg. Ann.* 66, 97 (1845).
[5] *Pogg. Ann* 150, 386 (1873).
[6] *Tonind Zeit* (1876), 272.
[7] *Sprechsaal* (1889), 153.

With increased heating, the feldspar for the most part melts and the quartz likewise goes into solution. With a further increase in burning temperature, a still smaller amount of feldspar and only the larger quartz grains are observed in a colorless groundmass. With high magnifications, a network of long, thin, weakly doubly refracting needles can be observed. Another burning does affect the constituents but little, there being more glass and gas-bubbles present. According to Hussak, therefore, porcelain consists of an amorphous groundmass with a few large quartz grains, a few large gas-bubbles and fine needles resembling sillimanite, Al_2O_3. SiO_2.

Vernadsky[8] heated refractory clay at white heat for 72 hours and observed small prismatic crystallites. These were optically positive, showed an extinction parallel to the prismatic axis and were not soluble in cold concentrated hydrofluoric acid. He observed similar but better developed crystallites by heating a mixture of alumina and silica in the molecular ratio of 1:2 to about 1400°. The needles often lay around unmolten alumina or silica. Isolated crystals analyzed 37.31 percent SiO_2 and 63.65 percent Al_2O_3, which agrees closely with the theoretical analytical figures for Al_2O_3. SiO_2 viz. 37.02 percent SiO_2 and 62.98 percent Al_2O_3. Vernadsky also found that the two other modifications of Al_2O_3. SiO_2, cyanite and andalusite changed to sillimanite at 1320°-1380°. The latter has also been observed by other investigators.

Mellor[9] observed crystallized sillimanite in a Chinese and Berlin porcelain and held the same views concerning its formation as Vernadsky.

Glasenapp[10] came to the following conclusions as a result of his microscopic studies. All clays dissociate at a sufficiently high temperature into a glassy groundmass and a crystalline portion. Their dissociation increases with increasing temperature and with duration of burning. He believed that the glassy groundmass was richer in silica and that the crystalline material was richer in alumina.

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[8] *Bull. de la Soc. fr. de Min.* 13, 256, (1890).
[9] *J. Soc. Chem. Ind.* **26**, 375 (1907).
[10] *Tonind. Ztg.* **31**, 1167 (1907).

Plenske[11] has investigated the microstructure and formation of porcelain. He divides porcelains microscopically as follows:

I. Porcelain without crystallites, in which he notes quartz grains of 2-30μ in diameter with sharp edges and faces, very little being dissolved. Kaolin has lost its crystal outline but occurs as an aggregate of curled up amorphous grains 2-6μ in diameter; gas bubbles are also noted.

II. Porcelain with very few isolated crystallites, which he notes in a glassy groundmass, amorphous $Al_2O_3.2SiO_2$, quartz, bubbles and prism crystallites showing double refraction, parallel extinction and positive optical character. They may attain 20μ in length. Only a small quantity of quartz is dissolved.

III. Porcelain with larger groups of crystals, consisting of a glassy groundmass with large groups of crystallites, cloudy amorphous $Al_2O_3.2\ SiO_2$, quartz more or less corroded with a zone of glass rich in silica. The crystallites occur irregularly, also grouped in parallel order and sometimes they show lattice structure.

IV. Porcelain with larger groups of crystallites. This type shows an equal distribution of many crystallites in a glassy groundmass. The quartz varies greatly in amount and is more or less corroded. Amorphous $Al_2O_3.2\ SiO_2$ is observed. According to Plenske the vitrification of porcelain proceeds as follows: The feldspar on fusion dissolves the kaolin and quartz, but more of the former than of the latter. The large quartz grains are dissolved at the edges. On cooling, the crystals of sillimanite are formed by devitrification while in places where all the quartz is dissolved, glass is noted as the result of cooling. Kaolin burned in the high glost fire appears amorphous with but few crystals due to the fluxing action of impurities. In kaolin-quartz bodies no mutual chemical reaction takes place. Quartz-feldspar mixtures show mutual solution. Kaolin-feldspar mixtures show amorphous $Al_2O_3.2\ SiO_2$ in cloudy form deposited in the feldspar glass, a phenomenon observed in porcelain. In part, solution has taken place with subsequent crystallization of sillimanite. The crystals of sillimanite appear only with a content of 50 percent clay sub-

[11] *Sprechsaal*, 41, No. 20, 21, 22. *Tonind Zeit.* 32, 1343-5 (1908).

stance and are best developed in 75 percent clay substance and 25 percent feldspar. With a ratio of 1 clay to 3 feldspar no devitrification takes place. The longer and higher the burn the greater is the number of crystals formed, excepting in those bodies high in feldspar burned at cone 9 or below. Porcelain thus appears to be hypocrystalline and prophyritic consisting of a glassy groundmass, amorphous $Al_2O_3.2\ SiO_2$, corroded crystals of quartz, sillimanite and cavities or bubbles.

A. Zoellner[12] made a painstaking study of porcelain. His work consisted in the preparation of porcelain bodies, their microscopic study and the separation of the crystalline portion by means of dilute hydrofluoric acid (30-35 percent). He found that feldspar at the temperature of the porcelain kiln may dissolve 3.5 percent alumina, 14 percent clay substance and 60-70 percent quartz. These relations are not changed in the presence of more than two components. Hard porcelain consists of a glassy groundmass filled with numerous needle-like crystals, corroded quartz and many air bubbles. The crystals agree optically with sillimanite. Porcelain burned at a temperature lower than cone 12 shows fused feldspar, partially attacked quartz and an amorphous silicate, probably dehydrated clay substance. The analysis of the residue from two porcelains and Seger porcelain, isolated with hydrofluoric acid, showed the molecular ratio of $SiO_2:Al_2O_3$ to be 1:1 confirming the optical evidence of the presence of sillimanite. All the sillimanite is derived from the kaolin or clay substance and is not due to devitrification (as Plenske held), but to a molecular change of the clay substance, an action which is accelerated by the presence of fluxes. Clay substance is thus split up into an amorphous, glassy silicate higher in silica and into the crystalline silicate higher in alumina, sillimanite. Optical or chemical examination offers a means of distinguishing between hard-burned and soft-burned porcelain, the formation of sillimanite being the criterion.

Watts[13] comes to the following conclusions: A hard spar will do no more than bond the other constituents at temperatures lower than cone 9. The clay substance is first dissolved in the

[12] *Sprechsaal*, 41, 471—3 continued and ending 533-6.
[13] *Trans. Am. Cer. Soc.*, 11, 185-201 (1909).

feldspar glass, and later the flint particles begin to be dissolved until at cone 10 the porcelain begins to show evidence of sillimanite crystallization. At cone 13, a hard spar becomes completely fused and is completely fluid permitting such crystallization as conditions and other ingredients will allow.

PRESENT INVESTIGATION

In order to obtain as much information as possible on the constitution and microstructure of porcelain, it was decided to investigate the changes involved in each of the raw materials by burning at various known temperatures, in systems consisting of two of the raw materials, and in what might be classed as a ternary system of the three raw materials, feldspar, quartz and kaolin. Furthermore, it was proposed to examine commercial specimens representing the technical practice in various countries.

Both the method of immersed grains and that of thin sections were used in the petrographic examinations. The latter method is too well known for extensive comment, and it suffices here to state that the thin sections were ground, prepared and mounted in the usual manner. This method was used only for photography and as an adjunct to the method of immersed grains to furnish data on the structural relationship of the constituents, but even here it was limited in the scope of its applicability, owing to the extreme minuteness and intergrown character of some of the constituents.

For the determination of the physical and chemical changes taking place in the burning of the specimens and to obtain reliable data concerning their constitution, the method of immersed grains was employed. It furnished information to some extent regarding structural relations, especially where observations had to be made, with high magnifications on poorly developed material. The method consisted of detaching a small piece of the specimen to be examined, tapping it to a powder of the desired fineness and immersing portions of the powder in liquids of known indices of refraction. The advantages of this method over that of thin sections in the study of artificial silicates has been repeatedly shown by the Geophysical Laboratory in their researches and by

our laboratory in the studies on the constitution and hydration of Portland cement.

A. Feldspar. The melting points and thermal relations of the feldspars have been thoroughly investigated. Orthoclase and microcline are used almost exclusively in the production of porcelain, although some of the plagioclases may occur as impurities in the feldspar raw material. According to many investigators orthoclase melts under 1200° to an apparently solid isotropic phase. The melting interval is not sharp but extends through quite a range of time. As the temperature is raised above the melting point, the viscosity of the isotropic phase is lowered and eventually at about 1300° the isotropic phase forms a thick viscous liquid which is extremely resistant to devitrification.

The plagioclases form a complete isomorphous series with albite and anorthite as end members. According to Bowen[14] albite melts at 1100° ± 10° and anorthite at 1550° ± 2°. He has also determined the solidus and liquidus temperatures for all the intermediate concentrations and the curves representing these temperatures are typical for completely isomorphous series.

B. Silica. Concerning tthe thermal properties of silica, Fenner[15] reports the following:

α quartz (stable at ordinary temperature) inverts to β quartz at 575°. At 870° ± 10° with the aid of sodium tungstate as a flux, β quartz inverts to β_2 tridymite, and with the same flux at 1470° ± 10° β_2 tridymite inverts to β cristobalite which melts at 1625°. With an absence of mineralizer and with fairly rapid cooling, β cristobalite does not revert to β_2 tridymite, nor does β_2 tridymite revert to β quartz. β cristobalite reverts readily to α cristobalite at about 180°-270° and β_2 tridymite reverts likewise to β_1 tridymite at 163°. The latter compound reverts to α tridymite at 117°. Time is a great factor in the quartz-tridymite and tridymite-cristobalite inversions. It is easily possible to heat quartz rapidly enough so that it will invert above 1400° at once to cristobalite with probably only a small quantity of tridymite. With the thermal behavior of feldspar and quartz well-established, it was thought unadvisable to repeat this work.

[14] Z. *Anorg. Chem.* 82, 283-307.
Am. J. Science, 35, 577-99 (1913).
[15] *J. Wash. Acad. Sci. II*, No. 20 (1912). *Am. Jr. Sc.* 36-331-84 (1913).

C. Kaolin. Kaolin is an inclusive term applied to compounds of the general type $Al_2O_3.2SiO_2.2H_2O$ (kaolinite). Some of the white burning clays approach the composition of pure kaolin very closely.

Kaolin is a material concerning whose behavior on heatting many and varied opinions have been held. It is known, for example, that the combined water is lost within a certain temperature range. Le Chatelier, Hillebrand, F. W. Clark, McNeil, Knote, Ashley, Mellor and Holdcroft, Rieke and others have found different endothermic ranges. Perhops the most authoritative investigation of this problem is that of Brown and Montgomery[16]. They found that the combined water is expelled at comparatively low temperatures, so that even at 450° the bulk of it may be driven off. In heating clay to constancy of weight at different temperatures, the velocity of the dehydrating reaction is very slow at first and becomes suddenly rapid at 500°. The remaining portion of the water is removed more slowly so that a temperature of not less than 800° is required to accomplish complete expulsion.

Furthermore, an exothermic energy change was noted at about 925° by Le Chatelier and by Ashley[17], and at about 900° by Mellor and Holdcroft[18]. From a study of the hygroscopicity, specific gravity, and solubilities in hydrochloric acid of clays heated to 600°-700°, Mellor and Holdcroft concluded that above 500° kaolin dissociates into free alumina, free silica and water. The exothermic change noted may be a physical change of the alumina. The formation of sillimanite at higher temperatures is then due to a recombination of a part of the free silica with alumina.

Sokoloff[19] from his study of the solubility in hydrochloric acid of clays heated to various temperatures, reached conclusions similar to those of Mellor and Holdcroft. According to Sokoloff the free alumina resulting from the dissociation of the kaolin may not be identical with aluminium hydroxide in pure condition.

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[16] *Bureau of Standards Tech. Paper No. 21* (1913).
[17] *J. Ind. Eng. Chem. 3*, 91-5 (1910).
[18] *Trans. Eng. Ceram. Soc.* 10, 94-120.
[19] *Tonind. Zeit 36*, 1107-1111.

In the present investigation two fairly pure kaolins were used, a washed North Carolina kaolin and an English kaolin. The chemical analyses were as follows:

	North Carolina. *percent.*	*English.* *percent.*
SiO_2	45.80	47.68
Al_2O_3	38.65	37.73
Fe_2O_3	.91	.70
CaO	.26	.16
MgO	.11	.46
Na_2O	.05	.06
K_2O	.23	.67
H_2O	14.00	12.55

The microscopic examination of the North Carolina kaolin showed that the chief constituent was a kaolinitic material which occurred in extremely small, flaky grains. These grains appeared mainly amorphous, although a low order gray interference color was observed on a few. Due, however, to the low birefringence and the minuteness of the grains, interference figures could not be obtained. The index of refraction was, however, about 1.55. In addition was noted quite a quantity of sericite, a smaller amount of fine-grained quartz and an extremely small amount of biotite in greenish brown flakes.

Kaolinitic material was the chief constituent of the English kaolin. It was noted in very fine flaky grains which, however, were larger than those of the North Carolina kaolin, and showed more of a tendency toward anisotropy. Indistinct biaxial negative interference figures were obtained and the mean index of refraction was found to be 1.55. Sericitic mica was observed in a larger amount than in the North Carolina material, but no biotite was noted. A small amount of quartz was observed which was coarser grained than the other constituents.

Both of these kaolins were heated in a platinum resistance furnace for 5 hours at 600° and for 2 hours at each of the following temperatures: 900°, 1100°, 1200°, 1300°, 1400°, 1450°

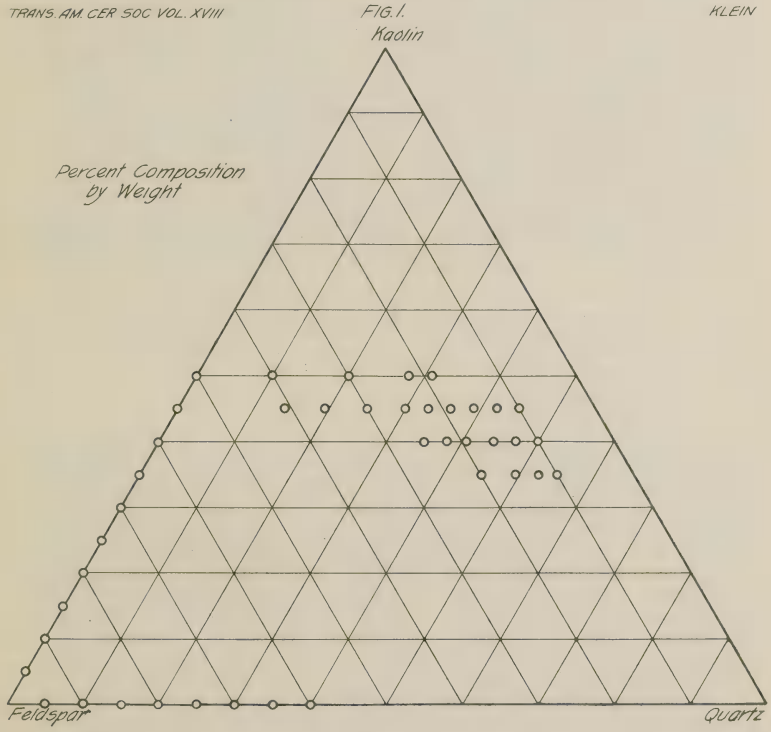
and 1500°. A microscopic examination was made of each of the burns.

The examination of these specimens revealed the following facts. As soon as the water of combination was driven off, the kaolinitic material became entirely isotropic, but the index of refraction altered to only a slight extent. Then there was no profound change noted in the optical properties until the temperature of burning reached upwards of 1200°. At this point, there was noted a beginning of an incipient dissociation of the kaolinitic material which became manifest by the formation in very small amounts of two isotropic substances, one of which had an index lower than 1.50 and the other had an index higher than 1.60. They were extremely intergrown and showed no attempt toward crystal form.

The dissociation remained incipient until a burning temperature of 1400° was reached, when a complete dissociation occurred and the two isotropic compounds noted above were formed. The higher refracting isotropic material showed no crystalline development, but occurred as extremely minute, almost submicroscopic, irregular to rounded grains intimately intergrown with the lower refracting product of dissociation. The effect was such that, due to reflection and refraction of light passing through them, these grains of dissociated kaolin appeared cloudy and brownish in color. In addition, there were noted a few aggregates of minute, thin, prismatic crystals. These were apparently isotropic being too thin to show distinct interference, and the index of refraction, which could not be obtained accurately, was greater than 1.60.

Between 1400° and 1450° nearly all the minute isotropic higher refracting grains assumed a prismatic development. Most of these minute needle crystallites were so thin as to be apparently isotropic, and their length was not over .01 mm. For the most part they exhibited no regular orientation, but occasionally they showed parallel grouping, and sometimes these parallel grouped aggregates intersected each other at an angle of 60°, giving rise to a lattice structure. Occasionally an aggregate of these needles showed a first order gray to white interference color. The extinction was then noted parallel to the

prism axis, and the character of the principal zone was positive. The index of refraction was about 1.64. Interference figures, although not often obtained, were biaxial, and the optical character was positive. The plane of the optic axis was parallel to the prism axis. These optical properties agree with those given for the mineral sillimanite, $Al_2O_3.SiO_2$.



The evidence points to the fact that these needle crystals result directly from the crystallization of the amorphous higher refracting compound noted in specimens burned at 1400°, since, firstly, the latter disappear with the formation of the needle crystallites. Secondly, in the specimens burned at 1450° a slight amount of amorphous high refracting material was noted from which minute needle crystals extended, showing this crystallization in an incipient state. It, therefore, seems certain that this

amorphous material is no more or less than an amorphous phase of sillimanite, and its formation results from the dissociation of the kaolin molecule before there is sufficient mobility for molecular orientation in any definite crystalline form.

D. Feldspar-Quartz Series. The feldspar used was a Maine feldspar with the following analysis:

SiO_2	69.56
Al_2O_3	17.36
Fe_2O_3	0.30
CaO	.22
MgO	.12
Na_2O	3.64
K_2O	6.90
	98.10

It consisted for the most part of orthoclase with a small amount of plagioclase having a composition about that of albite-oligoclase. In addition quartz and muscovite and microcline were noted in approximately equal amounts. A quantitative microscopical determination of the quartz and muscovite gave a value of ten percent, indicating the presence of about five percent quartz and five percent mica.

The following mixtures (see Fig 1) were prepared and burned at 1340° and at 1460°.

Mark	Feldspar *percent*	Quartz *percent*
F- 5	95	5
F-10	90	10
F-15	85	15
F-20	80	20
F-25	75	25
F-30	70	30
F-35	65	35
F-40	60	40
F-45	55	45
F-50	50	50

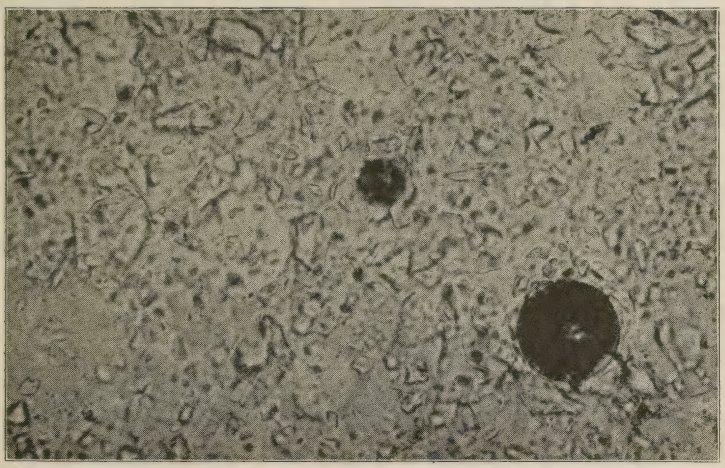


FIG. 2. Showing specimen with a composition of 90 percent feldspar, 10 percent quartz, burned at 1340°. Note the extremely small amount of quartz dissolved in the feldspar glass as evidenced by the amount of quartz present and the infrequent development of rounded, etched grains. This photograph was taken in ordinary light. Magnification = 300 diam.

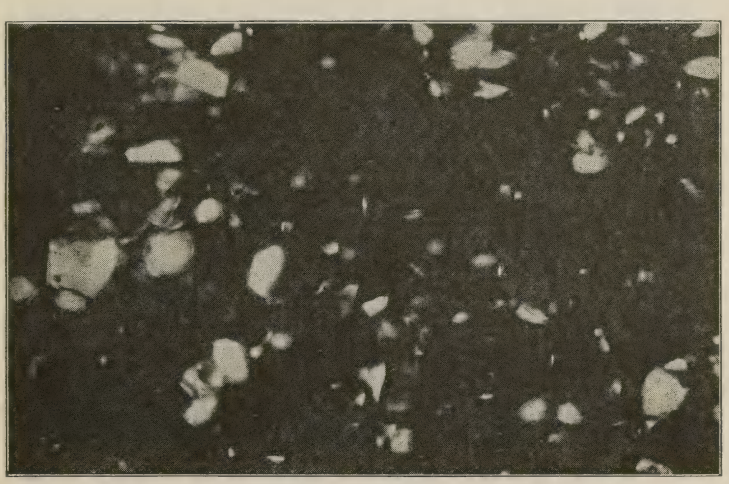


FIG. 3. Identical with Fig 2 except that this photograph was taken in plane polarized light, crossed nicols. Note the crystallinity of the quartz and the isotropy of the glass. Magnification = 300 diam.

The mixtures made up to the above percentages were placed in a refractory unglazed chemical porcelain crucible and heated in a large down-draft gas-fired kiln, using compressed air. The total time consumed in making a run was about 24 hours, and the kiln was kept at the desired temperature for 2-3 hours.

F-5 (1340°). The microscopic examination revealed the fact that the feldspar melted entirely to a glass and that the quartz was present as irregular grains showing more or less angularity, and but a small amount of solution in the feldspar glass, as was evidenced by a slight rounding of the corners and edges of the smaller grains. Quantitative microscopical determinations of the amount of undissolved quartz present gave an average of about nine percent. Since about five percent quartz was present originally and five percent was added, it can be seen that only a small amount of solution took place.

F-10 (1340°). This specimen showed exactly the same structure as F-5, but for the increased amount of quartz. Grains of the latter showed but little rounding and the quantitative microscopic determinations indicated that about fifteen percent was present.

F-15, etc. (1340°). These specimens resembled each other as well as F-5 and F-10 in constitution and structure. They differed from each other in the increasing quartz content, and the correspondingly decreasing feldspar glass content. In all cases the amount of quartz dissolved was small, and only the smaller grains showed slight but appreciable rounding due to solution. (See Figs. 2, 3, 4 and 5). The quantitative determinations of quartz present affirmed the statement that the solubility of the quartz at this temperature is comparatively small. F-40, which originally contained about forty-three percent quartz, gave an average of about forty percent present. Likewise, F-45, whose quartz content must originally have been about forty-eight percent, gave an average of about forty-five percent present.

F-5, etc. (1460°). — The quartz dissolved in the feldspar glass in all the specimens except F-40, F-45 and F-50. In these specimens there was a trace of quartz left which was noted in very small round grains, (See Fig. 6). For all practical purposes it may be said that quartz dissolves in a feldspar melt at

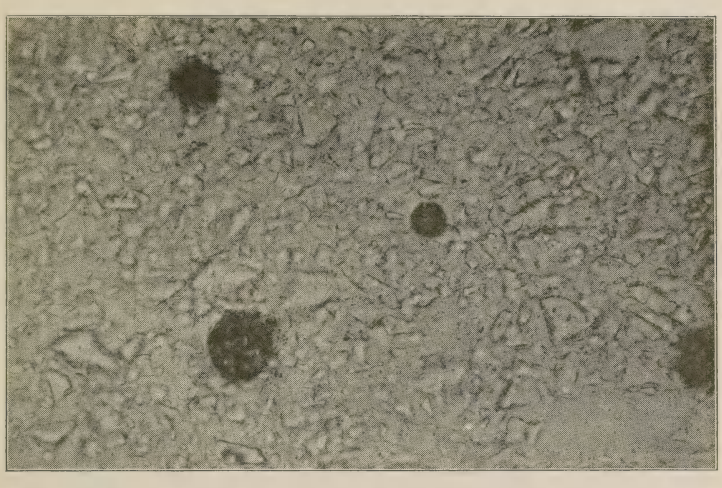


FIG. 4. Showing specimen consisting of 55 percent feldspar and 45 percent quartz burned at 1340°. Except for the additional quantity of quartz present, note the similarity to Fig. 2. This photograph was taken in ordinary light. Magnification = 300 diam.

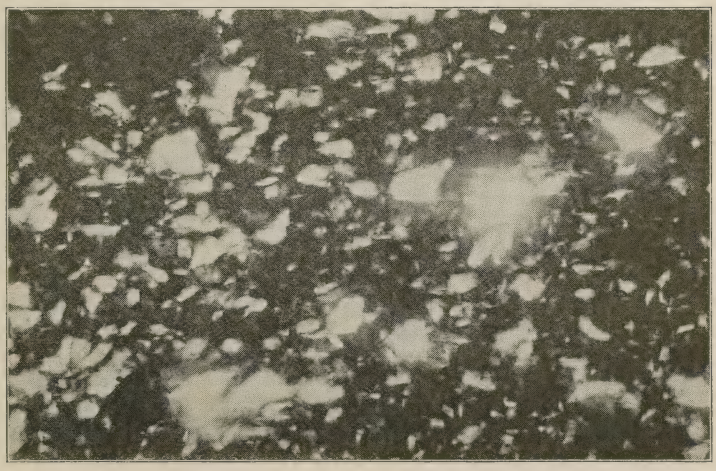


FIG. 5. This photograph is identical with Fig. 4 except that it was taken in plane polarized light, with crossed nicols. Note the increased amount of quartz here as compared with Fig. 3. Magnification = 300 diam.

or below 1460° at least at the concentration of fifty percent quartz to fifty percent feldspar.

E. Feldspar-Kaolin Series. The same Maine feldspar was used in this series as was used in the feldspar-quartz series. The kaolin was the washed North Caroline kaoliin described previously.

The following mixtures (see Fig 1) were prepared and burned at 1340° and at 1460°.

	Feldspar *percent*	Kaolin *percent*
C-5	95	5
C-10	90	10
C-15	85	15
C-20	80	20
C-25	75	25
C-30	70	30
C-35	65	35
C-40	60	40
C-45	55	45
C-50	50	50

The data on burning is identical with the data given on the burning of the feldspar-quartz series.

C-5 (1340°). In this specimen the feldspar melted entirely to a glass containing inclusions of quartz which was introduced as an impurity in the feldspar and kaolin raw materials and aluminium silicate. The quartz inclusions showed but little solution as indicated by a slight rounding of the sharp corners of the small grains, whereas the larger grains were angular. There appeared to be no profound difference in the refractive index between the glass adjacent to the quartz grains and the glass at a distance from them.

The original kaolinitic material disocciated entirely into silica and aluminium silicate. The former lost its entire identity in the feldspar melt, whereas the latter was observed in apparently isotropic aggregates of almost submicroscopic individuals whose index (greater than 1.60) was much higher than that of the glass.

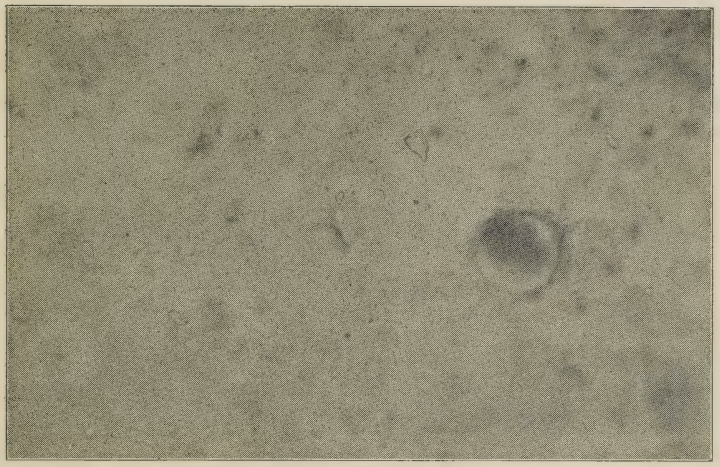


FIG. 6. Showing the specimen with a composition identical with that of Fig. 4, burned at 1460°. Note the last vestige of undissolved quartz grains. Magnification = 300 diam.

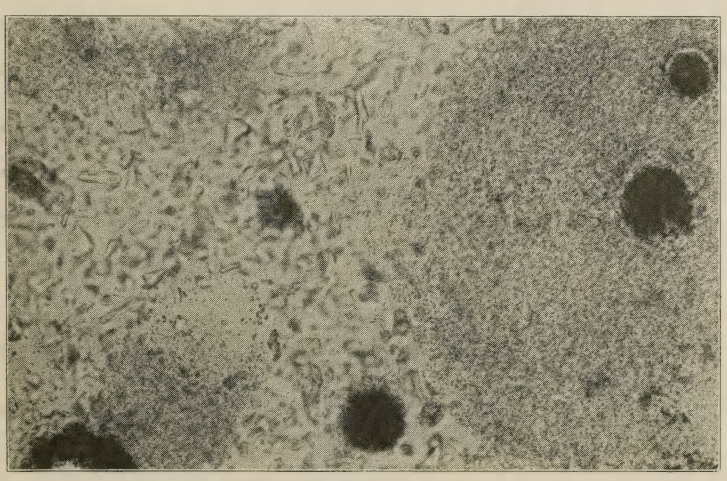


FIG. 7. Showing the feldspar-clay body containing ten percent clay and ninety percent feldspar. This body was burned at 1340°. Note the fine grained, non-crystalline appearance of the amorphous sillimanite, and the presence of undissolved quartz grains which entered as impurities in the feldspar and clay raw materials. Magnification = 360 diam.

If crystallization had taken place at all, the prismatic crystallites were less than .002 mm. in greatest dimension

C-10, etc. (1340°). — In all the specimens of the series the feldspar melted, and quartz dissolved in the feldspar glass to only a small degree. The kaolinitic material, however, differs somewhat in the various specimens. In C-10, the condition of the aluminium silicate was the same as in C-5; it was apparently amorphous sillimanite, (See Fig. 7). In C-15 a few sillimanite needle crystallites were observed, and the amount increased as the kaolin content increased at least up to the kaolin concentration of C-50.

C-5, (1460°). — Microscopically this specimen consisted of a homogeneous isotropic glass, showing neither quartz, amorphous sillimanite, nor sillimanite crystallites. Of particular interest was the complete solution in the feldspar glass of the small amount of aluminium silicate present at 1340°.

C-10, etc., (1460°). — In these specimens the quartz had practically all dissolved, since at most only a trace of minute rounded grains were observed. Sillimanite needles, (See Fig. 8), occurred in all the specimens in increasing amounts as the kaolin content increased. The needles were perfectly developed

TABLE I. ANALYSES OF MATERIALS USED IN THE PREPARATION OF FELDSPAR-QUARTZ-CLAY SPECIMENS

	NORTH CAROLINA KAOLIN	TENNESSEE BALL CLAY	GEORGIA KAOLIN	FELDSPAR	QUARTZ
	percent	*percent*	*percent*	*percent*	*percent*
Silica	45.84	50.80	45.69	70.79	99.23
Alumina	38.46	32.09	37.52	16.75	0.32
Ferric oxide	0.57	1.56	0.53	0.22	.10
Titanium oxide	.11	1.23	1.61	.....	.....
Calcium oxide	.19	0.18	.18	.24	.08
Magnesium oxide	.08	.40	.25	.18	.13
Sodium oxide	.07	.38	.11	3.29	.....
Potassium oxide	.35	1.14	.20	8.26	.....
Loss on ignition	14.44	12.49	14.05	.46	.22
Total	100.11	100.27	100.14	100.19	100.08

NOTE — In the feldspar-quartz-clay specimens, the clay was introduced as North Carolina kaolin, Tennessee ball clay and Georgia kaolin in the proportions of 5: 1: 1.

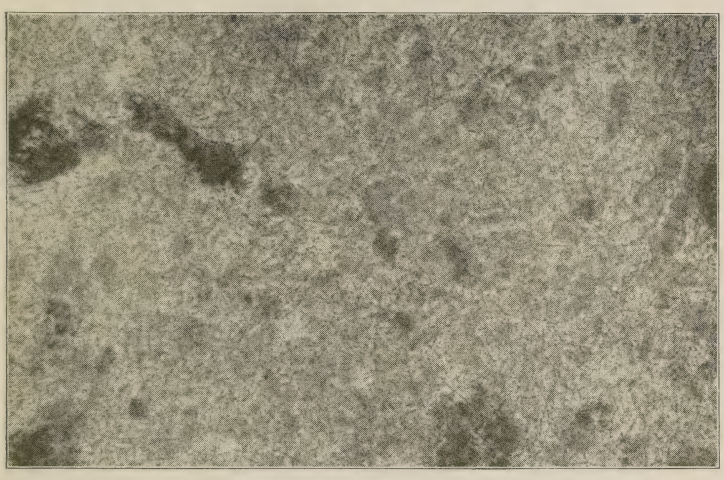


FIG. 8. Showing innumerable fine sillimanite needle crystallites developed by heating a body consisting of fifty percent clay and fifty percent feldspar to a temperature of 1460°. Magnification = 360 diam.

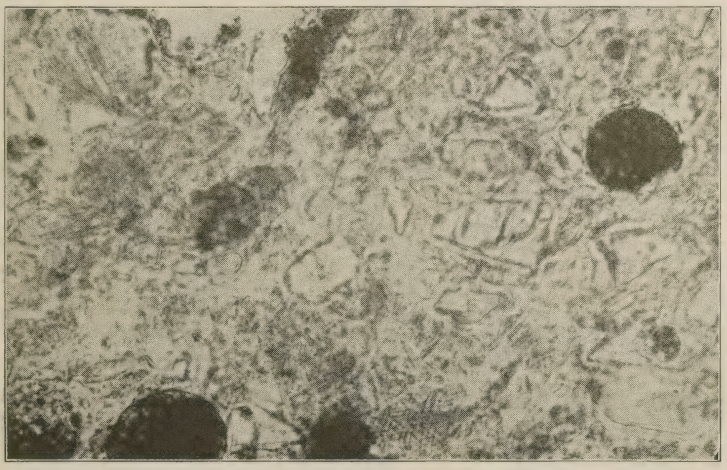


FIG. 9. Showing specimen No. 4 with a composition of 35 percent clay, 19 percent feldspar and 46 percent quartz. It was burned at 1400°. Note the presence of well developed acicular sillimanite crystals, in one instance showing the typical lattice structure. Note also the considerable quartz solution. Magnification = 500 diam.

and were comparatively large. For the most part they possessed no definite orientation, although occasionally the parallel grouping and the lattice structure described previously were noted.

F. Feldspar-Quartz-Clay Bodies. 1. First series (high quartz, low feldspar). — The specimens of this series were prepared by Bleininger and Teetor in their study on the viscosity of porcelain bodies[30]. The data on their composition and burning will be given here but briefly since it may be found in full detail in the report of their work.

The compositions are indicated in Table II and Fig. 1. These must be regarded as approximate to a degree, since they do not represent the analyses of the burned specimens but are calculated from the analyses of the raw materials given in Table I. It may be noted that these bodies are comparatively high in quartz content and low in that of feldspar. Some in fact approach in composition that of typical whiteware bodies. The clay used was a mixture of five parts of North Carolina kaolin, one part Georgia kaolin and one part Tennessee ball clay. The firing of all the specimens for the viscosity investigation was carried to the following temperatures: 1065°, 1100°, 1125°, 1160°, 1190°, 1220°, 1250°, 1280°, 1310°, so that each specimen examined microscopically was subjected to nine different burnings between 1065° and 1310°. Furthermore, these specimens after having been examined, were reburned at 1400° for observation concerning the changes involved between 1310° and 1400°.

A glance at Table II will indicate that series I consists of 4 sub-series. In each sub-series the clay content has been kept constant and the feldspar varied with the quartz. Thus the sub-series are as follows: 35 percent clay, 10-19 percent feldspar and 55-46 percent quartz; 40 percent clay, 10-25 percent feldspar and 50-35 percent quartz; 45 percent clay, 10-25 percent feldspar and 45-30 percent quartz; 50 percent clay, 19-22 percent feldspar and 31-28 percent quartz. The microscopic observations will be given collectively for each sub-series.

Specimens 1-4, (1310°). — In general these specimens closely resembled each other in the changes undergone by the

[30] *Bureau of Standards, Technical Paper No. 80* (1914).

raw materials and in the development of new constituents. The most important constituent observed was quartz. This mineral was noted in angular grains of variable size which showed practically no evidence of solution. The amount of quartz decreased from Nos. 1 to 4. Both the clay and the feldspar lost their original identity. The latter melted to a low refracting glass which increased in amount from Nos. 1 to 4. The clay dissociated almost entirely into silica and aluminium silicate. The silica dissolved in the feldspar glass, but the aluminium silicate was apparently not soluble at this temperature. It was noted, (1) mainly as amorphous sillimanite in minute rounded isotropic inclusions in the glassy groundmass, and was distinguished from the groundmass by its index of refraction which was greater than 1.60; (2) as a small quantity of true sillimanite occurring in extremely fine needle crystals about .01 mm. in length. These were mainly apparently isotropic. Aggregates of these needles showing double refraction could be observed; the extinction was then noted parallel to the prism axis, the axis of elongation of the needles. The character of the principal zone was positive, and the mean index of refraction was found to be about 1.64. The ratio of the amount of crystallized to amorphous sillimanite did not vary in specimens 1, 2, 3 and 4.

Specimens 5-10, (1310°). — These specimens resembled each other microscopically. The original quartz was noted unchanged, and the angularity of the grains was proof that practically no solution had taken place. The amount of quartz decreased from specimen 5 to specimen 10. Both the feldspar and the clay lost their original identity. The feldspar melted to a low refracting glass which dissolved the silica formed by the complete dissociation of the clay. The other dissociation product of the clay, aluminium silicate, was observed both as amorphous sillimanite and crystallized needles of sillimanite. As in specimens 1-4, the amount of the latter was much smaller than that of the former. Crystallites were very minute and often times could be observed with only the highest magnifications.

Specimens 11-16, (1310°). — The data on the microscopipal examination is very nearly the same for each specimen. The original feldspar melted entirely, and the amount of feldspar

glass increased from specimen 11 to specimen 16. The content of quartz decreased from specimen 11 to specimen 16, but the character of the quartz grains were identical, and the amount of solution in the feldspar glass was practically nil. The clay showed complete dissociation with the formation of silica, amorphous and crystallized sillimanite. The silica dissolved completely in the glass. The sillimanite was noted mainly as amorphous sillimanite with a very small amount of fine needle crystals, most of which were too minute to indicate birefringence.

Specimens 17-18, (1310°). — The microscopical examination of these specimens revealed a similarity to the previous specimens. The complete melting of the feldspar was noted, as well as the complete dissociation of the clay with the formation of much amorphous sillimanite and a little crystallized sillimanite in very fine needles. As observed previously the quartz showed practically no solution.

Specimens 1 to 4, (1400°). — These specimens, (See Fig. 9), showed fundamental differences from those burned at 1320°. A considerable decrease was noted in the amount of free quartz, brought about by its solution in the glass. The solution was, however, by no means complete. The residual quartz grains were much rounded and etched. The solvent action proceeded more rapidly along the edges and along the fracture cracks of the quartz resulting in rounded grains and in aggregates of rounded grains containing interstitial glass. The amount of residual quartz decreased from specimen 1 to specimen 4.

Another fundamental change noted was the great increase in number and, to a lesser degree, in size of the sillimanite needle crystallites, accompanied by a corresponding decrease in the amount of rounded inclusions. The tendency toward crystallization was noted by the attempts at elongation of some of the remaining amorphous sillimanite grains. These assumed a club-like form which could be observed only with higher magnification.

The amount of glass increased from specimen 1 to specimen 4. Moreover owing to the increased solution of quartz, the amount of glass in each of the specimens was greater than the amount in the specimens burned at 1310°.

TABLE II. PORCELAIN SERIES I

SPECIMEN NO.	CLAY	FELDSPAR	QUARTZ	SiO_2	Al_2O_3	Fe_2O_3	TiO_2	CaO	MgO	Na_2O	K_2O
	percent	*percent*	*percent*	*percent*	*percent*	*percent*	*percent*	*percent*	*percent*	*percent*	*percent*
1	35	10	55	82.04	15.71	0.36	0.18	0.14	0.15	0.39	1.02
2	35	13	52	81.13	16.23	.36	.18	.15	.15	.49	1.28
3	35	16	49	80.24	16.74	.37	.18	.16	.15	.60	1.55
4	35	19	46	79.34	17.26	.37	.18	.17	.15	.69	1.81
5	40	10	50	79.87	17.79	.39	.20	.15	.15	.40	1.06
6	40	13	47	78.96	18.31	.40	.20	.16	.15	.50	1.33
7	40	16	44	78.05	18.83	.40	.20	.17	.16	.61	1.59
8	40	19	41	77.14	19.35	.40	.20	.17	.15	.71	1.86
9	40	22	38	76.24	19.88	.41	.20	.17	.16	.82	2.12
10	40	25	35	75.34	20.40	.41	.20	.18	.17	.92	2.39
11	45	10	45	77.63	19.88	.43	.24	.16	.16	.41	1.09
12	45	13	42	76.73	20.41	.43	.24	.16	.15	.51	1.36
13	45	16	39	75.82	20.93	.44	.24	.17	.16	.62	1.62
14	45	19	36	74.90	21.46	.44	.24	.18	.16	.72	1.89
15	45	22	33	73.99	22.00	.44	.24	.18	.16	.82	2.16
16	45	25	30	73.08	22.52	.45	.24	.18	.17	.93	2.43
17	50	19	31	72.65	23.63	.47	.26	.17	.15	.73	1.93
18	50	22	28	71.72	24.16	.48	.26	.17	.15	.84	2.20

NOTE — The above analyses were calculated to a water-free basis from the analyses of the raw materials.

Specimens 5 to 10, (1400°). — The changes involved in raising the burning temperatures from 1310° to 1400° were identical with those observed in specimens 1-4. Quartz showed a considerable decrease in quantity, and the residual quartz grains bore evidence of solution, *viz.*, rounding and etching of the grains. The amount of quartz decreased from specimen 5 to specimen 10.

Sillimanite needles were noted in much greater amount than the amorphous sillimanite. These were in most instances very minute, but occasionally individuals were noted 20μ in length. They occurred mainly in irregular aggregates, but aggregates showing parallel grouping and the lattice structure described previously were also noted. The small crystallites were apparently isotropic while the larger ones sometimes showed the first order yellow interference color. Extinction parallel to the axis of elongation was noted, and the principal zone was positive. Biaxial interference figures could occasionally be observed with a positive optical character, and the plane of the optic axis was parallel to the axis of elongation. Quantitative values for the indices of the α, β and γ rays could not be obtained because the crystallites were much intergrown with glass from which these could not be completely separated. The approximate mean index was found to be slightly above 1.64. The optical properties establish the compound definitely as sillimanite, $Al_2O_3\,SiO_2$. The amount of glass increased from 5 to 10.

Specimens 11 to 16, (1400°). — The report of the microscopical examination of specimens 11 to 16 is similar to that of the preceeding sub-series. The considerable decrease in the amount of quartz with the corresponding increase in the quantity of glass was noted. The quartz grains were rounded and etched. They showed solution along the edges and along the fracture cracks.

Observations on the growth of the amount and to a lesser degree of the size of sillimanite needles were made. The development of sillimanite between 1300° and 1400° was similar to that observed with the previous specimens of Series I. The incipient formation of sillimanite needles from amorphous sillimanite was also noted in the elongation of the rounded grains

TABLE III. PORCELAIN SERIES II

SPECIMEN NO.	CLAY	FELDSPAR	QUARTZ	SiO_2	Al_2O_3	Fe_2O_3	TiO_2	CaO	MgO	Na_2O	K_2O
	percent	*percent*	*percent*	*percent*	*percent*	*percent*	*percent*	*percent*	*percent*	*percent*	*percent*
1	35	30	35	76.15	19.16	0.38	0.18	0.18	0.16	1.10	2.77
2	35	40	25	73.08	20.93	.39	.18	.20	.16	1.43	3.65
3	35	50	15	70.08	22.63	.41	.18	.21	.17	1.78	4.50
4	35	60	5	67.07	24.36	.42	.18	.22	.18	2.18	5.38
5	35	65	.....	65.55	25.22	.42	.18	.24	.18	2.30	5.81
6	40	40	20	70.81	23.01	.43	.20	.21	.17	1.45	3.70
7	45	30	25	71.56	23.42	.45	.24	.19	.16	1.11	2.87
8	45	35	20	70.13	24.31	.46	.24	.20	.17	1.28	3.31
9	45	40	15	68.60	25.17	.47	.24	.21	.17	1.47	3.75
10	45	55	.....	64.08	27.82	.48	.24	.24	.18	1.99	5.08
11	50	30	20	69.28	25.56	.50	.26	.19	.16	1.13	2.91
12	50	40	10	66.20	27.34	.50	.26	.22	.16	1.49	3.81
13	50	50	.....	63.15	29.12	.52	.26	.23	.17	1.84	4.68

NOTE — The above analyses were calculated to a water-free basis from the analyses of the raw materials.

to form a club-like grain and in the cementing or agglomeration of a number of these rounded inclusions to form a prism-like crystal.

Specimens 17 and 18, (1400°). — The observations on 17 and 18 burned at 1400° are similar to those of the preceding specimens burned at this temperature. The amount of quartz noted was considerably less than that noted at 1300°, and the grains showed evidence of much solution. A corresponding increase in the amount of glass was also noted. The sillimanite needles while greatly in excess of the extremely small amount of amorphous sillimanite showed no better needle crystal development than in the previous specimens of Series I where the quantity of original clay was smaller.

(2) Second series (low quartz, high feldspar). The specimens of this series were prepared and used by Bleininger and Kinnison in their study on the viscosity of porcelain bodies[21]. As with Series 1, the data on the composition and burning will be touched on but briefly here, since it is given in fuller detail in the report of their investigation.

The compositions are indicated in Table III and Fig. 1. The figures in this table do not represent the true analysis of the burned specimens but are only approximate since they were calculated from the analyses of the same raw materials employed in Series 1 and given in Table I. From the data on composition, it is evident that some of these specimens are identical with typical porcelain and floor tile bodies. The firing of all specimens with the exception of Nos. 2, 4 and 5 was carried to 1275°, 1295°, 1315°, 1345°, and 1380°. The maximum temperature attained with specimens Nos. 2 and 5 was 1345°, and with No. 4 was 1315°.

From a consideration of Table III, it is seen that the series may be sub-divided into four sub-series in which the clay content is constant and the feldspar varies with the quartz. These are as follows: 35 percent clay, 30-65 percent feldspar, 35-0 percent quartz; 40 percent clay, 40 percent feldspar, 20 percent quartz; 45 percent clay, 30-55 percent feldspar, 25-0 per-

[21] *Trans. Am. Cer. Soc.*, 17, 130-6 (191).

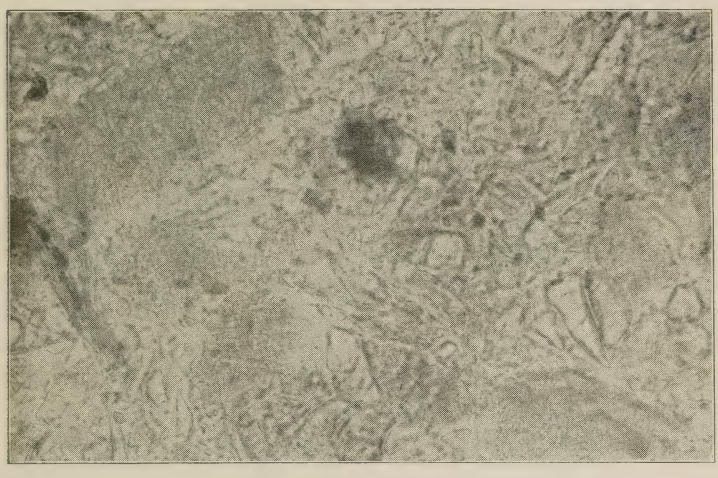


FIG. 10. Showing specimen No. 1 with a composition of 35 percent clay, 30 percent feldspar and 35 percent quartz. It was burned at 1380°. Note the crystalline sillimanite development and the evidence of considerable quartz solution. Magnification = 380 diam.

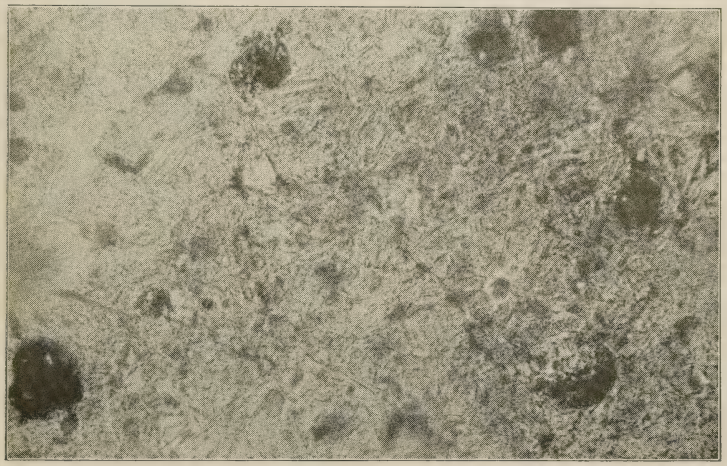


FIG. 11. Showing specimen No. 13 with a composition of 50 percent feldspar and 50 percent clay, burned to 1380°. Note the development of sillimanite needles and the presence of an extremely small amount of residual quartz in mainly elliptical grains. Magnification = 300 diam.

cent quartz; 50 percent clay, 30-50 percent feldspar, 20-0 percent quartz.

Specimens 1-5 — All the specimens of this sub-series cannot be compared with each other since the maximum burning temperature was 1315° in the case of No. 4, 1345° in the case of Nos. 2 and 5, and 1380° in the case of Nos. 1 and 3.

The microscopic examination of specimen No. 4 revealed the complete melting of the feldspar to an isotropic glass. The quartz bore evidence of only a slight solution as attested by the incipient rounding of the edges especially of the smaller grains. Extremely fine sillimanite needles were noted, as was also amorphous sillimanite. The needles were so minute as to be observed with only higher powers and only in a few instances could even the faintest interference colors be seen.

The complete melting of the feldspar and the solution of quite a little quartz was observed in specimens Nos. 2 and 5. Not all the quartz had disappeared, even in No. 5 where the quartz present occurred only as an impurity in the clay and in the feldspar, a very small amount being noted in more or less rounded grains. Sillimanite needles appeared to be more plentiful than in No. 4 and slightly better developed, whereas amorphous sillimanite occurred in smaller amount.

Considerable solution of quartz was observed in specimens No. 1, (See Fig. 10), and 3, the latter containing an extremely small amount. No amorphous sillimanite was noted since it was changed completely to the crystallized form. Although they appeared to be better developed than those of Nos. 2, 4 and 5, the major portion of the needle crystals was still very minute. Optical properties when obtained agreed with those given for sillimanite.

Specimen 6 — The final burning of specimens Nos. 6 to 13 reached a maximum temperature of 1380°. Specimen No. 6 showed considerable solution of the quartz in the glass. There was no amorphous sillimanite noted, and the needle crystals were for the most part minute and irregularly intertwined.

Specimens 7-10. — Considerable solution of the quartz was also noted in the specimens of this sub-series. Specimen No. 9 contained only a very small amount of residual quartz observed

in rounded grains showing profound solution. Number 10 contained only a trace of quartz. Sillimanite was noted in thin acicular crystallites which occasionally showed parallel grouping and lattice structure, although they were mainly irregularly intergrown. The amount of glass increased from No. 7 to 10 with a corresponding decrease in the amount of quartz, while the amount and development of the sillimanite in each showed no appreciable differences.

Specimens 11-13. — The conditions obtaining in this sub-series were analogous to those in the previous sub-series. Solution of the quartz took place to a considerable degree, and the residual quartz grains were very much etched and rounded. That complete solution did not take place was evidenced by a trace of quartz noted in specimen No. 13, where it occurred only as an impurity in the feldspar and clay raw materials, (See Fig. 11).

Practically no amorphous sillimanite was noted. The sillimanite crystallites varied in size from minute needles showing apparent isotropy to some about 50 μ in length in which first order interference colors were observed and which agreed with the mineral sillimanite in the determinable optical properties.

G. Pores, Communicating and Sealed. In all the specimens examined in this investigation bubbles and blebs have been noted more or less regularly. No especially close observations have been made on these, since most of them were destroyed in the preparation of the specimens for examination which was effected by tapping to a fine powder. In general our observations have been that a porous structure with communicating pores in greater amount than sealed pores is present in bodies burned at lower temperatures (*e. g.*, the temperature of burning of whiteware bodies). With increase in burning temperature, the communicating pores decrease and the sealed pores increase, but with still further increase in burning temperature *e. g.*, in hard fired-porcelain bodies, the sealed pores also decrease, although no body was examined in which they had totally disappeared.

H. Results Obtained From the Study of Laboratory Prepared Bodies. Kaolin suffers little optical change with heating up to 1200°. At this temperature the grains appear to be

homogeneous, isotropic, with an index slightly higher than that given for the mineral kaolin. Above this temperature a slow incipient dissociation occurs up to about 1400° when the material is entirely dissociated into what appears to be amorphous silica and amorphous sillimanite. With heating to 1450° the latter gives rise to characteristic needle crystals of sillimanite. When a flux *e. g.,* feldspar is present, this dissociation proceeds at lower temperatures, and the silica loses its identity by solution in the feldspar melt. Thus at 1340°, kaolin is entirely dissociated with the formation of both amorphous and crystalline sillimanite and with still higher temperature the amorphous modification crystallizes entirely, and the sillimanite crystals develop in size.

The investigation of the feldspar-quartz series shows that quartz is but slightly soluble in a feldspar melt at 1340°, but that with increase in temperature solution proceeds to a greater extent and that at 1460° in melts which originally contained fifty percent quartz, there exists no more than a trace of undissolved quartz.

The study of the clay-quartz-feldspar series reveals a number of interesting facts. The feldspar is the first constituent to melt. It then acts as a flux-solvent in the solution of the other constituents. In both high quartz-low feldspar and low quartz-high feldspar bodies, burned at 1310°, the original clay dissociates into both amorphous and crystalline sillimanite, the former greatly predominating. The sillimanite needles are so extremely small that often they appear isotropic with crossed nicols. Quartz is only slightly soluble at this temperature and the quartz grains show distinct angularity with little evidence of solution along the edges.

At higher temperatures (1380°-1400°) the amorphous sillimanite disappears and gives rise to crystallized sillimanite. These crystals appear to grow somewhat in length and thickness, and oftentimes parallel grouping and lattice structure are noted. Quartz, also, dissolves to a considerable extent, and the residual grains of this constituent show much rounding of the corners and edges. In fact the tendency is toward elliptical and spherical grains.

TABLE IV. TEMPERATURE OF PORCELAIN BURNING AS INDICATED BY THE CONSTITUTION AND MICROSTRUCTURE OF THE BURNED SPECIMENS

OBSERVATIONS ON CONSTITUTION AND MICROSTRUCTURE	PROBABLE BURNING TEMPERATURE
Feldspar melted to a glass, no apparent dissociation of dehydrated clay; no solution of quartz.....	About 1200°
Feldspar melted; slight dissociation of dehydrated clay with the formation of amorphous sillimanite; no solution of quartz.....	About 1225°
Feldspar melted, increase in dissociation of dehydrated clay with formation of amorphous sillimanite; no solution of quartz.....	About 1250°
Feldspar melted; considerable dissociation of dehydrated clay with the formation of amorphous sillimanite; no solution of quartz.....	About 1275°
Feldspar melted; almost complete dissociation of dehydrated clay but the amount of sillimanite crystals are still very small; no solution of quartz	About 1300°
Feldspar melted; complete dissociation of dehydrated clay with an increase in amount of sillimanite crystals although amorphous sillimanite still predominates; beginning of quartz solution	About 1325°
Feldspar melted; sillimanite needles appear to be almost as plentiful as amorphous sillimanite; solution of quartz is still comparatively small although noticeably greater than at 1325°.....	About 1350°
Feldspar melted; amount of sillimanite needles present greater than that of amorphous sillimanite; quartz dissolved to quite an extent....	About 1375°
Feldspar melted; dissociation product of dehydrated clay consists entirely of crystallized sillimanite; quartz considerably dissolved..	About 1400°
Feldspar melted; comparatively little undissolved quartz; porcelain consist almost entirely of glass and sillimanite needle crystals.....	About 1425° or more

At this point we are led to a discussion of the question of the comparative solubility of kaolin and quartz in feldspar. Such a question is in the light of this investigation manifestly an indefinite one. What is meant by the solubility of kaolin? It has been definitely shown that kaolin dissociates into silica and aluminium silicate. It has been indicated also that the resultant silica is soluble in the feldspar glass, but that at the maximum temperature attained in this investigation, 1460°, the aluminium silicate is comparatively little soluble. Therefore to consider the solution of kaolin in feldspar at temperatures of porcelain burning is to consider an action which does not take place. However, if the criterion of the action of feldspar on clay is to be the solution in the feldspar melt of the silica resulting from the dissociation of the kaolin, then such a comparison can be readily made. Unqualifiedly it is as follows: Kaolin is more soluble in feldspar than quartz under conditions of temperature and time analogous to those of porcelain practice.

I. The Temperature of Burning—Constitution and Microstructure Scale. As stated previously it was hoped that there was a fairly definite relationship between the burning temperatures of porcelain bodies and the physical and chemical changes taking place in the raw materials and noted in the microscopical examination of the finished product. That such is generally the case may be seen from the results of the study of the feldspar-quartz, feldspar-clay, and feldspar-quartz-clay series. Therefore, relying on such a relationship the burning temperatures of the commercial bodies were predicted from the microscopical examination. The actual burning temperatures were not known to the author, but Prof A. V. Bleininger possessed the complete data, and a comparison of both showed that the predicted temperature of burning was remarkably close to the measured temperature. In fact an accuracy within 25° C. was maintained in nearly all cases. The scale is given in Table IV.

J. Commercial Bodies. This series embraces bodies ranging from a whiteware body to a high-fired hard porcelain body. The technical practices of many countries are represented in order to determine the essential differences in constitution and microstructure effected in bodies of the same commercial class

by dissimilar conditions of burning. The practices of the following countries are represented: United States, England, Germany, France, Austria and Denmark.

Whiteware, Ohio. — The following constituents were noted: feldspar glass, dehydrated clay and quartz. The feldspar appeared to have entirely melted to a glass. The dehydrated clay was noted as amorphous grains which showed practically no evidence of dissociation into lower refracting silica and higher refracting amorphous sillimanite. The quartz grains were noted in considerable amount. They varied in size from almost submicroscopic grains to grains measuring .5 mm. across and showed absolutely no evidence of solution in the feldspar glass. The melting of the feldspar and the absence of dissociation of the clay was evidence of the fact that the burning temperature of this body was not over 1200°.

Whiteware Body (Unknown Brand). — The microscopical examination revealed the following facts. The feldspar had entirely melted to a glass acting as a cement for the other constituents. Nearly all the dehydrated clay was unchanged. That an extremely small part did dissociate was evidenced by the presence of minute rounded amorphous sillimanite grains of higher index than the other constituents occurring as inclusions in the dehydrated clay grains and in the glass. Quartz was present in considerable quantities, but the grains showed no evidence of solution. The melting of the feldspar and slight dissociation of the dehydrated clay indicated a burning temperature of 1200°-1225°.

Whiteware — J and G Meakin Hanley, Eng. — The feldspar melted to a glass acting as a binder for the clay and quartz. The observations on the clay were very unsatisfactory. There was considerable undissociated dehydrated clay present, but some of it appeared cloudy and brownish in color. The microscope did not resolve anything further of the structure of these brownish grains so that this condition is probably due to the presence of amorphous sillimanite grains which are just submicroscopic in size. The quartz while not so plentiful as noted in the two American whiteware bodies was found in sharp cor-

nered grains giving no evidence of solution. The indications were of a burning temperature between 1200° and 1225°.

Vitrified Ware, West Virginia. — A considerable dissociation of the dehydrated clay was noted with the formation of minute rounded isotropic grains of amorphous sillimanite and a trace of extremely fine needles of sillimanite. The silica resulting from the dissociation lost its identity in the feldspar glass. Quartz was present in a considerable quantity and showed absolutely no evidences of solution. The partial dissociation of the clay indicates a temperature of burning of about 1250°.

Vitrified Ware, New York. — The examination of this specimen showed that the feldspar had melted completely. The dehydrated clay had dissociated to a considerable extent to amorphous sillimanite mainly with only a trace of minute sillimanite needles. A large quantity of quartz was noted, and the quartz grains bore no evidence of solution, (See Fig. 12). The constitution of this specimen indicated a burning temperature of about 1250°.

Sanitary Body, New Jersey. — The microscopical evidence in this body was analogous to that of the two preceding specimens. The feldspar had melted completely to a low refracting glass. The quartz grains showed no apparent solution. The dehydrated clay showed considerable dissociation into amorphous sillimanite and an extremely small amount of fine sillimanite needles, all of which indicates a burning temperature of about 1250°.

Electrical Porcelain, Pennsylvania. — The important feature of this specimen, (See Fig. 13), was the almost complete dissociation of the dehydrated clay with the formation of considerable amorphous sillimanite, and a much less amount of minute sillimanite crystallites. There was only a very small quantity of undissociated dehydrated clay present. The quartz grains showed only a trace of solution, if any at all, and were sharp cornered in nearly every instance. The feldspar had melted entirely to a glass. The almost complete dissociation of the dehydrated clay, the great preponderance of the minute rounded amorphous sillimanite grains over the sillimanite needle crystal-

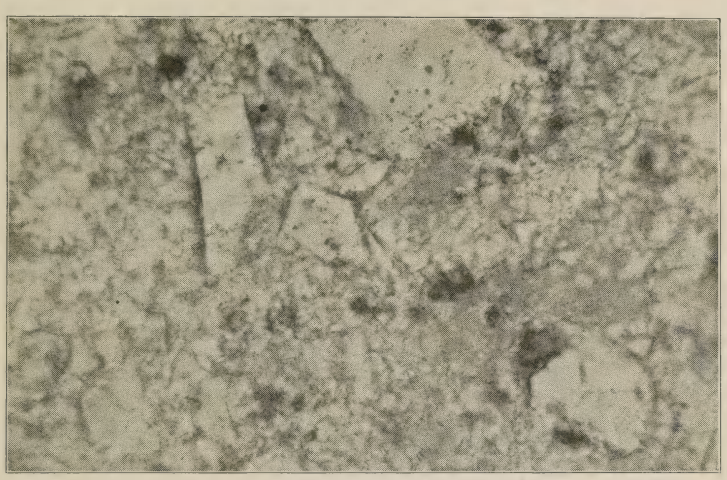


FIG. 12. Showing a specimen of vitrified ware manufactured in New York. Note the absence of sillimanite needle development and the presence in the groundmass of cloudy material whose structure cannot be resolved in a thin section but which is due to the presence of minute amorphous sillimanite. Note also the large quartz grains showing absolutely no solution. Magnification = 300 diam.

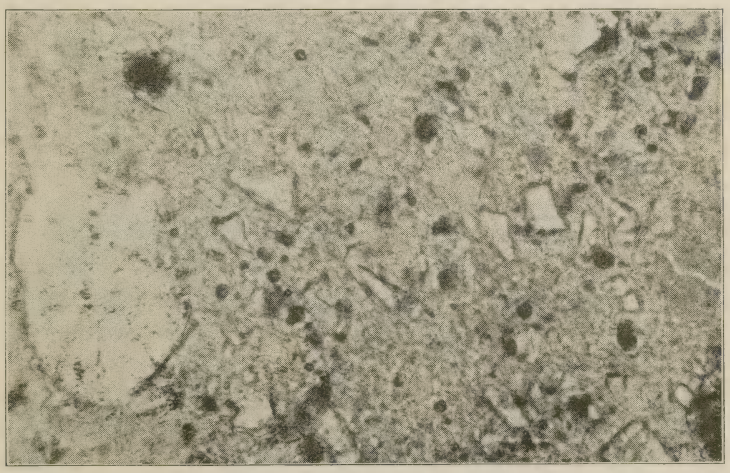


FIG. 13. Showing a specimen of electrical porcelain manufactured in Pennsylvania. Note the variation in size of the quartz grains and the absence of any evidence of quartz solution. The very small amount of crystalline sillimanite cannot be definitely seen in the photograph but the cloudiness of the groundmass, due to amorphous sillimanite, may be readily recognized. Magnification = 300 diam.

lites and the absence of any decided solution of quartz indicates a burning temperature of 1250° to 1275°.

Spark Plug Body. — The following observations were made on this body. A low refracting glass resulting from the melting of the feldspar was observed. The dissociation of the dehydrated clay had proceeded to a great extent with the formation of amorphous sillimanite and sillimanite needle crystallites of which the former greatly predominated. Quartz was present in considerable amount and appeared to have undergone no solution in the larger grains and a doubtful trace of solution in the smaller ones. The microscopical evidence here was identical with that of the previous specimen, and the burning temperature appears, therefore, to be about 1275°.

Hübschenreythe, Bavarian Porcelain. — At best but a slight solution of quartz in the feldspar glass was noted in this specimen. The grains possessed sharp edges, and evidences of slight solution were not general, being noted mainly in the smaller quartz grains. While there was little if any undissociated dehydrated clay present, the main dissociation product was amorphous sillimanite in minute rounded grains. A small amount of crystallized sillimanite was also present. The almost complete dissociation of the dehydrated clay together with the preponderance of amorphous over crystallized sillimanite points to a burning temperature of about 1300°.

Austrian Porcelain, Elsa. — The examination of this specimen revealed the complete dissociation of the dehydrated clay and the resultant formation of both amorphous and crystalline sillimanite, the former occurring in larger amounts. A large quantity of quartz was present and very slight evidence of solution was noted. The above data indicates a burning temperature of about 1300°-1325°.

Chemical Porcelain. — Bureau of Standards Body No. 10.— Complete dissociation of the dehydrated clay was observed in this specimen. The resultant sillimanite occurred in both the crystalline and amorphous form with the former as plentiful as the latter. The acicular crystallites of sillimanite were minute, extremely intergrown and in nearly all cases they were apparently isotropic. The quartz grains gave evidence of a small amount

of solution in the feldspar glass. That a greater degree of solubility was attained here than in the specimens burned at 1300°-1325° was shown by a more universal etching and a more pronounced rounding of the quartz grains. When this examination was made the author was unaware of the burning temperature of the body but from the above data he predicted a burning temperature of 1325°-1350°. Subsequently, he was informed by Mr. G. H. Brown of the Bureau who prepared and burned the body that the temperature of burning was 1350°.

Royal Meissen, Onion Pattern. — The microscopical examination of this body, (See Fig. 14), revealed a microstructure which was practically identical with that of the previous body. The dissociation of the clay had taken place with the formation of mainly very minute needles of sillimanite and rounded minute grains of amorphous sillimanite. The quartz content was fairly low, but the appearance of the individual quartz grains indicated only a small but appreciable solution. The burning temperature seems to be close to that of the chemical porcelain body No. 10, about 1325°-1350°.

Ahrenfeldt; Limoges France Porcelain. — As in the cases of the preceding specimen, the dehydrated clay had completely dissociated, but in this instance the dissociation had resulted for the most part in the formation of fine sillimanite needles and only a small amount of amorphous sillimanite. The quartz appeared to have undergone quite considerable solution in the glass, the etching and rounding of the grains being more pronounced than in the previous specimen. The evidence points to a burning temperature of about 1375°.

Theodore Haviland, Limoges, France Porcelain. — The dehydrated clay in this speciment had not only dissociated but the amorphous sillimanite resulting from the dissociation had practically all crystallized into needle crystals of sillimanite. These varied in size being more generally very small. Occasionally, however, they grew to a length of .1 mm. and when they attained this size, they exhibited anisotropy, parallel extinction, positive principal zone, biaxial positive interference figures; all of which are observed on sillimanite. The needles occurred more often in irregular aggregates, but at times parallel grouping and the

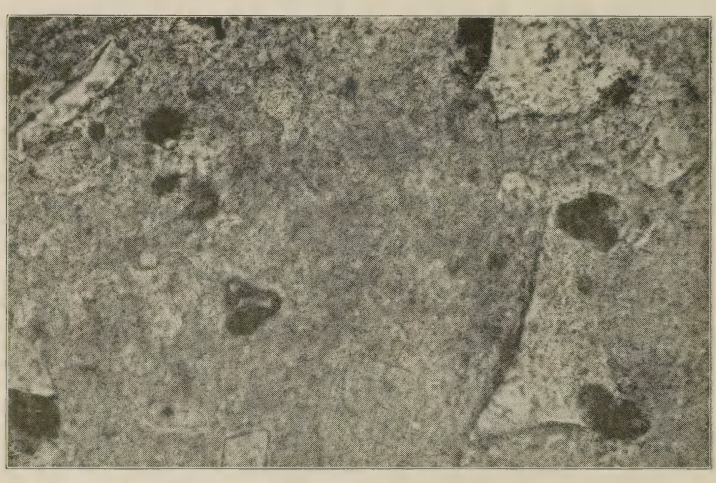


FIG. 14. Showing specimen of Meissen China; Onion Pattern. Note the presence of both crystallized and amorphous sillimanite. Only the smaller quartz grains shows evidence of solution as manifested by the rounding of the grains while the larger ones still retain their sharp corners. Magnification = 300 diam.

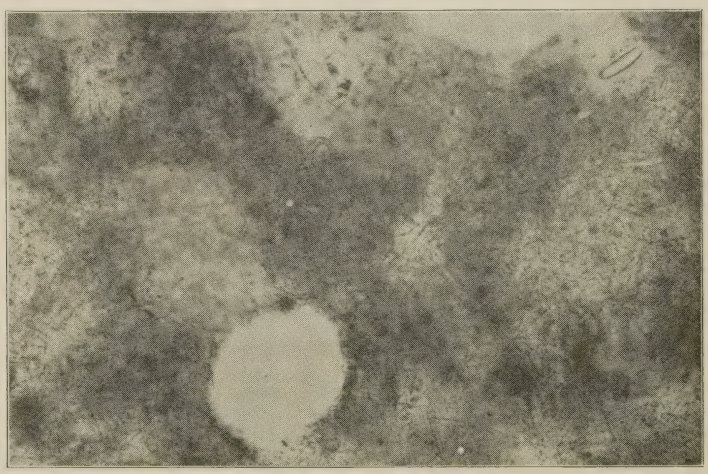


FIG. 15. Showing Haviland Limoges China; Ransom Plain Pattern. Note the development of sillimanite crystallites and the elliptical quartz grains, remnants of profound solution in the feldspar glass. Magnification = 440 diam.

lattice structure previously described, were observed. Quartz occurred in comparatively small amounts as rounded, sometimes almost spherical, grains. Aggregates of rounded grains were noted which were originally a single grain but solution had taken place along fracture cracks and had disintegrated the large grain into smaller ones. That a considerable solution of quartz in the feldspar glass occurred is very evident. The crystalline sillimanite development and the solution of the quartz indicates a burning temperature of about 1375° to 1400°.

Haviland; Limoges France Porcelain, Ransom Plain.—This specimen, (See Fig. 15), showed an interesting constitution and microstructure typical of high spar, hard fired porcelains. The constituents in order of importance were glass, sillimanite crystallites and quartz. The sillimanite occurred in acicular crystallites. Parallel grouping and lattice structure were sometimes observed although most of the crystallite aggregates possessed no regular orientation. Some of the needles were developed sufficiently so that first order yellow interference colors were observed with crossed nicols. The remaining optical properties agreed with those given for sillimanite. There were comparatively few minute rounded isotropic grains of amorphous sillimanite noted. The amount of undissolved quartz was not large, and the rounded to spherical appearance of the grains indicated the great progress which solution in the feldspar glass had made. The better development of sillimanite and evidence of greater solution of the quartz than in the Ahrenfeldt and Theodore Haviland bodies, was taken to indicate a slightly greater burning temperature probably about 1400°.

Royal Berlin Chemical Porcelain. — The microscopical examination of this body, (See Fig. 16). as well as the succeeding chemical porcelain bodies revealed even a higher burning temperature than that of the Limoges practice. The most important constituent observed was the glass resulting from the melting of the feldspar and the subsequent solution in the melt of silica resulting from the dissociated clay and of quartz. The sillimanite crystallites were much intergrown and varied in size from almost sub-microscopic needles to some which measured .1 mm. or more. The residual quartz grains were present in very small

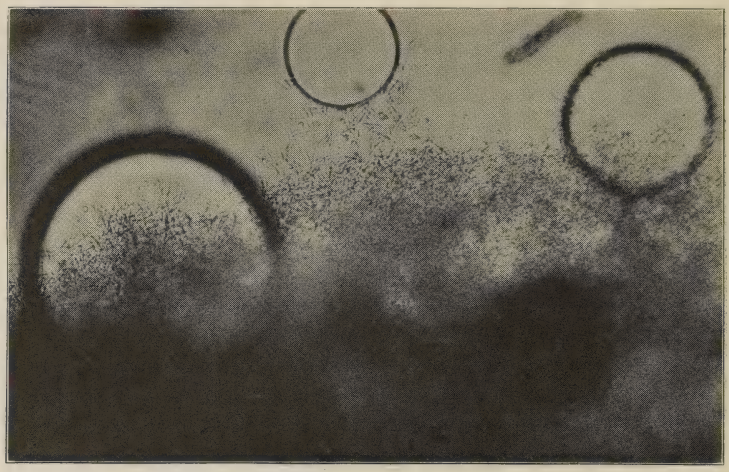


FIG. 16. Showing fracture piece of Royal Berlin chemical porcelain consisting of both body and glaze and demonstrating the reaction between them during the glost burn. Note the spherical blebs and the sillimanite needles extending from the body into the glaze. Because of the thickness of the fracture piece, the body appears opaque. Magnification = 300 diam.

FIG. 17. Showing a fracture piece of Thuringien chemical porcelain consisting of both body and glaze. Note the similarity to Fig. 16, except for the larger sillimanite needles. Magnification = 190 diam.

amounts, and their elliptical to spherical outline bore witness to the profound solution they had undergone. The increase in solution of quartz over that of the Limoges bodies pointed to a probable burning temperature of 1400°-1425°.

Thuringien Chemical Porcelain. — This body resembles the Royal Berlin body in constitution and microstructure, (See Fig. 17). The constituents in order of importance were, glass, sillimanite crystallites and undissolved quartz. The crystals of sillimanite ranged in size from extremely minute needles to those upwards of .1 mm. in length, and the optical properties of the largest needles which showed unmistakable anisotropy agreed with those given for sillimanite. Only a small amount of quartz was present, and the grains bore evidence of great solution. The similarity between the observations here and those of the previous specimen pointed to a burning temperature of 1400°-1425°.

Royal Meissen Chemical Porcelain. — This body resembled both the Thuringien and Royal Berlin bodies, (See Figs. 18, 19 and 20). The sillimanite crystallites attained a maximum length of .23 mm. and oftentimes exhibited parallel grouping and lattice structure. As may be expected, the quartz grains bore the earmarks of much solution. Oftentimes the residual quartz grains were surrounded by a zone of glass free from sillimanite crystallites, but almost as often these two constituents were intimately associated with each other. This observation was made not only with the Meissen body but with all the hard fired procelains examined where the quartz showed considerable solution. The above observations indicated a burning temperature of 1400°-1425°.

Bureau of Standards Chemical Porcelain No. 14. — The constituents of this body were a low refracting glass, sillimanite crystallites and residual quartz grains. The needles of sillimanite were very plentiful and for the most part very minute and intergrown. The amount of unchanged quartz was small (not over five percent). The burning temperature was predicted as being very close to that of the last three chemical porcelains examined, *viz.*, 1400°-1425°. The actual burning temperature proved to be 1425°. Of interest showing the great solution of the quartz at

FIG. 18. Showing a fracture fragment of Royal Meissen chemical porcelain. Note the similarity to Fig. 17. Magnification = 300 diam.

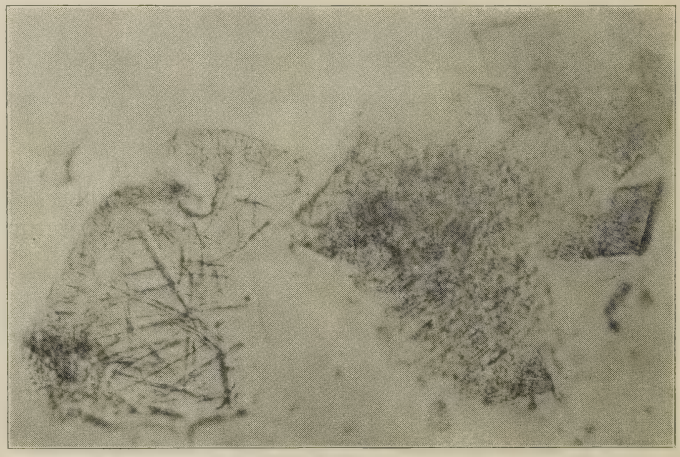


FIG. 19. Showing a fracture piece of Royal Meissen chemical porcelain body magnified to a higher degree than that of Fig. 18. Note the excellent sillimanite development. Magnification = 600 diam.

this temperature is its composition: clay fifty-five percent feldspar twenty percent and quartz twenty-five percent.

Royal Copenhagen Chemical Porcelain.,— This body proved to be the highest burned body examined. The following constituents were noted, glass, sillimanite needles and hardly more than a trace of quartz. The amount of sillimanite appeared to be greater than was observed in the other chemical porcelains. The crystal development, however, was not particularly remarkable. In fact almost all the sillimanite needles were very minute and

FIG. 20. Showing a fracture piece of Royal Meissen chemical porcelain consisting of body and glaze. This photograph is interesting in that it shows not only the sillimanite needle development between body and glaze and the blebs but also a grain of quartz exhibiting typical evidence of solution around the edges and along fracture cracks. Magnification = 190 diam.

exceedingly intergrown. The criterion for the burning temperature was quartz, or to be more exact the almost complete absence of quartz. The few quartz grains which were observed were for the most part so minute as to appear isotropic with crossed nicols and could be recognized only by the indices of refraction. There were some quartz grains present, however, which were unmistakably uniaxial, positive, but these also were much rounded and etched, due to solution. The evidence pointed to a burning temperature higher than 1425°.

K. The Relation of Microstructure and Constitution to Time of Burning. From the data on the investigation of bodies prepared in this laboratory and of commercial bodies it has been noted that there are marked differences in microstructure and constitution with differences in burning temperatures. The question now arises are there not great differences in constitution entailed in the ordinary differences in time of burning since this factor may vary in the practices of individual concerns? Undoubtedly there are some differences but these are relatively small when compared with changes involved with the variation of the burning temperature. For instance, the Bureau of Standards chemical porcelain body No. 14 was burned at 1425°, and the Royal Berlin chemical porcelain was burned at cones 15-16, about the same temperature. The data on the microstructure and constitution of these two bodies are practically identical, notwithstanding the fact that the burning of our body lasted about twenty-four hours from beginning to completion, the kiln being kept for three hours at the maximum temperature, whereas in European practice the time of maintaining the highest temperature range may be as long as twelve hours.

Another comparison may be made between chemical porcelain body No. 14 and the specimens of the feldspar-quartz-clay, Series I, which had received ten successive burnings from 1065° to 1400°. The constitution of the latter bodies notwithstanding their excessive burning, indicated a slightly lower burning temperature than the former. The quartz, for instance, did not show quite the effect of solution in the feldspar glass in any of the specimens of Series I as in body No. 14. This fact is indeed fortunate from the view point of predicting the burning temperature from the microscopical examination of the burned product. If there were great differences in constitution caused by small differences in duration of burning, a low temperature body burned for a longer period would resemble a high temperature body burned for a shorter period. Fortunately this is not the case.

L. The Absence of Cristobalite and Tridymite from Hard Porcelain Bodies.—As has been mentioned previously, quartz inverts to tridymite at 875° and to cristobalite at 1475° in the

presence of sodium tungstate as a flux. Furthermore, we have observed[22], in the burning of silica brick, the change of quartz direct to cristobalite at higher temperatures in the presence of only a slight amount of flux. Recently we have also noted the change of cristobalite to tridymite when the silica brick have been subjected, many times, to high temperatures under 1475°. Why then should we not observe these modifications instead of the residual quartz grains? The determination of cristobalite and tridymite in a porcelain is a matter of difficulty since these two compounds have such a low double refraction as to appear isotropic or nearly so in small grains and furthermore, the glass noted in porcelain varies in index depending upon its composition, although its index is always in the vicinity of the indices of these two compounds. A thorough and painstaking search has thus far revealed no definite appearance of cristobalite and tridymite, while the presence of residual quartz has been demonstrated in all cases. It is probable, therefore, that solution of the quartz in the glassy constituent takes place more rapidly than its inversion to cristobalite and to tridymite. Otherwise we ought to find unmistakable evidence of the presence of these compounds.

SUMMARY AND CONCLUSIONS

The investigation consisted in the microscopic petrographic examination of clay, feldspar-quartz, feldspar-clay and feldspar-quartz-clay bodies burned at various known temperatures. Furthermore, commercial bodies of various grades ranging from whiteware to hard fired chemical porcelain were investigated. The end in view was to obtain data as to the constitution and microstructure of porcelains and the changes in these involved by burning at various temperatures. Subsequently it was found possible to correlate to a degree, the constitution and microstructure with the burning temperature of bodies whose composition lay within the limits of whiteware and hard fired porcelains.

The result of this investigation leads to the following conclusions: clay appears homogeneous microscopically when

[22] *Met. Chem. Eng.* 13, 861-6 (1915).

heated up to 1200°. At about this temperature a trace of dissociation occurs. As the temperature is raised above 1200° the dissociation increases very slowly at first, then at an increasing rate until at 1400° it seems to be complete. The products of dissociation are silica and aluminium silicate. The latter compound has been identified as an amorphous phase of sillimanite from the following facts: it shows no crystalline form, has an index of refraction approximately above 1.60, and by heating at a higher temperature (about 1450°) it inverts to minute needle crystallites corresponding to sillimanite in all determinable optical properties.

Up to 1340° in mixtures of quartz and feldspar, the quartz dissolves to only a small extent in the feldspar glass. At 1460° the quartz is practically completely dissolved in specimens having as high a quartz content as fifty percent quartz to fifty percent feldspar.

At 1340° in specimens containing clay and feldspar, the clay dissociates entirely. The amount of crystallized and amorphous sillimanite increases with an increased content of clay at least to a concentration of fifty percent clay to fifty percent feldspar.

At 1460°, apparently ten percent clay is entirely soluble in the feldspar glass. With higher concentrations of clay the amount of crystallized sillimanite increases. The needle crystals are well developed and comparatively large.

At 1310° in quartz-clay-feldspar bodies, the feldspar is present as a glass; the clay shows almost complete dissociation with the formation of amorphous sillimanite mainly and but little crystallized sillimanite, while the quartz is undissolved and the grains may attain considerable size, up to .2 mm.

By burning these bodies at 1380°-1400°, the feldspar glass dissolves considerable quartz, there being only a comparatively small amount of residual quartz remaining. The quartz grains are much rounded and etched, and they seldom attain a length of .06 mm. The clay is dissociated with the formation of crystallized sillimanite although an extremely small amount of amorphous sillimanite may be present.

The changes involved by burning commercial bodies are identical with those of laboratory prepared bodies. Commercial ware ranges from a low burned porous whiteware in which except for the dehydration of the clay, only the feldspar is changed, to high fired chemical porcelain which consists of glass, sillimanite crystallites and more or less of residual quartz. The quartz grains observed in the whiteware and in the low fired vitreous ware are large and angular, attaining a length of .2 mm. or more, whereas in the hard porcelains, due to solution, the quartz grains are rounded, etched, and seldom exceed .05 mm. in length.

The constitution and microstructure of porcelain depend upon the temperature of burning and change as this temperature changes. Their variation with temperature is given in Table IV. This table has served as a basis for the prediction of the probable burning temperatures of the commercial bodies, a fact which was accomplished with success. It appears that the time of burning factor is by no means as important, as that of the burning temperature in determining the constitution and microstructure of the ware.

Mention must be made here that no cristobalite or tridymite has been definitely observed in any of the laboratory or commercial bodies examined. It appears that the quartz dissolves in the feldspar glass more readily than it inverts to the other modifications.

In conclusion we may state that the petrographic microscopic study of porcelain led to interesting and, it is to be hoped, important technical results. It has placed the chemical and physical processes involved in the formation of porcelain on a more quantitative thermal basis. Furthermore it has offered a means of predicting the burning temperature of ware from the examination of a fragment much too small in size to be satisfactory for even a chemical analysis.

Acknowledgments are due to Prof. A. V. Bleininger for his kindly suggestions throughout the work, to H. A. Brady for the preparation of the thin sections, and to W. L. Howat, C. S. Kinnison, and P. Teetor for the preparation of the bodies examined.

A STUDY OF BRISTOL GLAZES COMPOUNDED ON THE NORM BASIS

BY ARTHUR S. WATTS

The work of Prof. Staley in Vol. XIII, page 126, these *Transactions,* in which he exploits the use of "norms" as a basis of classification of the constituents of ceramic compounds, and the fact that the classification of glaze ingredients into "norms" furnishes a means of reducing the study to a three component system appealed to me so strongly that I determined to conduct such an investigation.

Regarding the actual development of "norms" in Bristol glazes, I am not prepared to speak, but it seems probable that when the glaze is fired to maturity, the laws governing selective crystallization or segregation should be operative at least to a limited degree. I am not attempting to prove the development of "norms" in Bristol glazes, but merely employing this as a means of subdividing the ingredients into three members from which the study may be developed.

The ordinary Bristol glaze contains the following chemical constituents introduced in the form noted:

K_2O and Na_2O introduced in feldspars.
CaO generally introduced as whiting or pulverized marble.
ZnO introduced as zinc oxide.
Al_2O_3 introduced in feldspar or kaolin.
SiO_2 introduced in feldspar, kaolin or as free silica.

The minerals which would normally form from Bristol glazes, cooled under the most favorable conditions, are microcline, albite, anorthite, wollastonite, willemite, and sillimanite.

As commercial feldspar is generally a mixture of microcline and albite, the most fusible mixture of these is employed. Sillimanite would not be expected to form, according to Iddings (Igneous Rocks, Vol. 1, pages 435-441), until after all the other

minerals named, and hence need not be considered in a preliminary study.

The normal minerals which therefore remain to be considered in a Bristol glaze composition are microcline, albite, anorthite, wollastonite, and willemite. Feldspar is the only member which is used in mineral form in Bristol glazes, the other "norms" are synthetic mixtures which have the same chemical compositions as the minerals.

The synthetic compositions of anorthite and wollastonite are worthy of special consideration since anorthite ($CaO\text{-}Al_2O_3\text{-}2\ SiO_2$) consists of one molecule each of calcium carbonate and clay substance, while wollastonite ($CaO\text{-}SiO_2$) consists of one molecule each of calcium carbonate and flint. The difference in their behavior in the glaze would therefore be an indicator of the difference produced in a glaze by the substitution of clay substance for free silica.

Willemite as a mineral is written $2\ ZnO\text{-}SiO_2$, but to maintain RO unity in this study which is ceramic practice, it will be considered as consisting of one molecule of zinc oxide and one-half molecule silica; *i. e.*, $ZnO\text{-}.5\ SiO_2$.

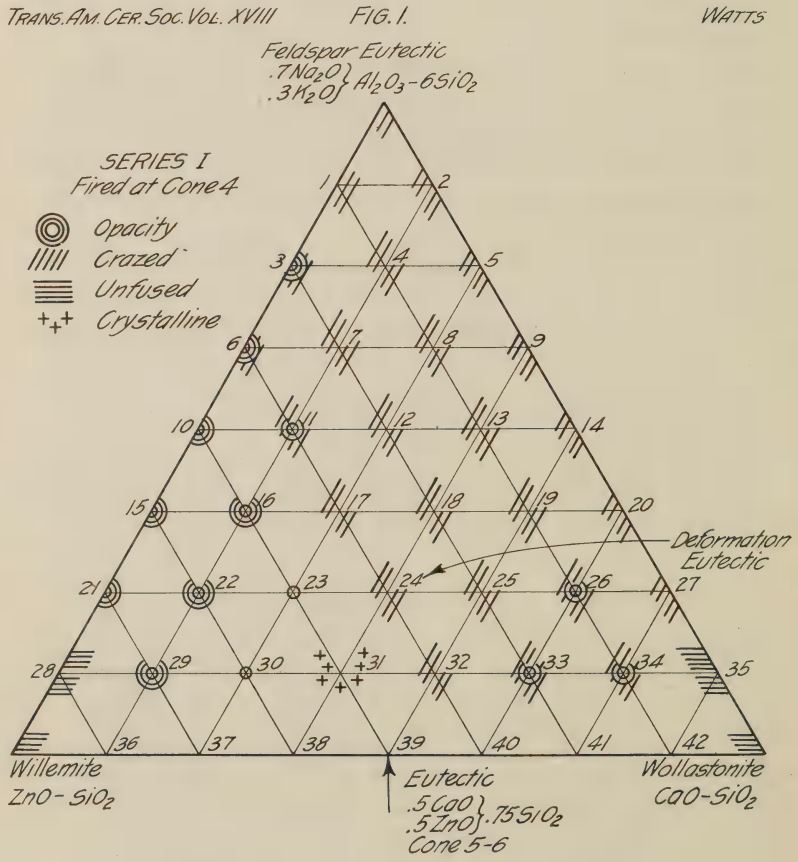
TABLE I. ANALYSIS OF MATERIALS EMPLOYED IN THIS STUDY

	FELDSPAR		WHITING	ZINC OXIDE	KAOLIN	FLINT
	POTASH	SODA				
SiO_2	71.75	65.5	1.30	....	45.50	99.40
Al_2O_3	16.70	21.17	2.14	....	39.30	.20
Fe_2O_3	.14	.18	.10	....	.05	.05
CaO	.25	.87	54.40	....	.....	.....
MgO	.....	1.40	.....	....	.....	.....
K_2O	8.59	1.00	.....	....	.42	.....
Na_2O	2.99	8.98	.....	....	.44	.....
Ign. loss	.35	.15	.....	....	14.20	.30
CO_2	.....	.....	42.68	....	.....	.....
ZnO	.....	.....	.....	98.9	.....	.....
Totals	99.77	99.25	100.62	98.9	99.91	99.95

The study is divided into two series of 45 members each, namely:

Series I. Feldspar-willemite-wollastonite and 42 intermediate members.

Series II. Feldspar-willemite-anorthite and 42 intermediate members.



Each end member was prepared by wet grinding together, for five hours, the necessary ingredients. The solid content of each mixture was determined, and the intermediate members produced by wet blending on a molecular basis. Each member thus

prepared was further mixed by hand for several minutes. The glazes were applied to dry unburned stoneware trials, and an attempt was made to secure a coating of equal thickness on all trials.

The intermediate members of each series were produced by blending on a molecular basis, therefore the chemical formula of any member of a series may be quickly determined by interpolating between the end members. For example Series I, member 11, consists of 50 percent feldspar, 12½ percent wollastonite norm, and 37½ percent willemite norm. It has the formula

$$\left.\begin{matrix}.500\ KNaO\\ .125\ CaO\\ .375\ ZnO\end{matrix}\right\}\ .5\ Al_2O_3\text{-}\ 3.3125\ SiO_2$$

STUDY OF SERIES I

Fusibility. The temperature of deformation of the end members of this series were as follows:

Feldspar eutectic-Cone 6, approximately 1250 degrees C.

Willemite norm-Cone 13, approximately 1390 degrees C.

Wollastonite norm-Cone 8, approximately 1290 degrees C.

The binary eutectics were as follows:

Feldspar-willemite eutectic-Series I. Member 3.

Formula:

$$\left.\begin{matrix}.525\ Na_2O\\ .225\ K_2O\\ .25\ \ ZnO\end{matrix}\right\}\ .75\ Al_2O_3\text{-}4.625\ SiO_2$$

Deformation temperature Cone 2, approximately 1170 degrees C.

No feldspar-wollastonite eutectic could be located.

Willemite-wollastonite eutectic-Series I, Member 39.

Formula:

$$\left.\begin{matrix}.5\ CaO\\ .5\ ZnO\end{matrix}\right\}\ .75\ SiO_2$$

Deformation temperature Cone 5 and 6, approximately 1240 degrees C.

The ternary eutectic of this series is Series I, Member 24.

Formula:

$$\left.\begin{matrix} .175\ Na_2O \\ .075\ K_2O \\ .375\ CaO \\ .375\ ZnO \end{matrix}\right\} .25\ Al_2O_3\text{-}2.06\ SiO_2$$

Deformation temperature Cone 1, approximately 1150 degrees C.

Opacity. The opacity of this series is difficult to indicate since the feldspar-wollastonite members of the series are immature while the feldspar-willemite members are in various stages of solution. The willemite-wollastonite members show immaturity toward the end members, increased fluidity from the wollastonite end of the series, and when the proportions are 1 to 1 a clear glass is produced. Beyond this and toward the willemite end of the series, crystals appear in increasing amounts until the glaze is supersaturated and becomes a dense mat, ending in immaturity.

In general the glazes of this series do not show the opacifying effect of the willemite norm unless the feldspar content is less than .5 equivalents, the wollastonite norm is less than .375 equivalents and the willemite norm exceeds .25 equivalents.

Opacity is first evidenced by the appearance of crystals which increase in size with increased feldspar content, and in amount with increased willemite norm contents.

Crazing. The tendency to craze is apparent whenever the feldspar content exceeds .25 equivalents or the willemite norm content is less than .375 equivalent.

Surface Texture. At cone 4, those members approaching the composition of the ternary eutectic have a glassy brilliant surface, and with increased heat treatment the range of composition producing glassy surfaces increases. Toward the willemite corner of the series the development of a satin-like texture is apparent. Toward the wollastonite corner of the series there is an abrupt change from a glassy surface to an earthy surface indicative of immaturity. Toward the feldspar corner of the series the gradual increase of viscosity due to increased Al_2O_3 content is evidenced by entrapped bubbles of air and by pin-holing.

STUDY OF SERIES II

Fusibility. The anorthite norm is the only one not used in Series I, and this has a deformation temperature of about cone 18.

The feldspar-willemite eutectic is the same as in Series I.

The feldspar-anorthite eutectic, if one exists, is

$$\left.\begin{matrix} .525\ Na_2O \\ .225\ K_2O \\ .25\ \ CaO \end{matrix}\right\} Al_2O_3\text{-}5.\ SiO_2$$

The willemite-anorthite eutectic is

$$\left.\begin{matrix} .25\ CaO \\ .75\ ZnO \end{matrix}\right\} .25\ \ Al_2O_3\text{-}.875\ \ SiO_2$$

and it has a deformation temperature of Cone 5, approximately 1230 degrees C.

The lowest deformation member of this series is Series II, Member 3, and this is also the binary feldspar-willemite eutectic.

This portion or the series was made into cones and tested, but no addition of anorthite norm covered by this study produced a lower fusing mixture.

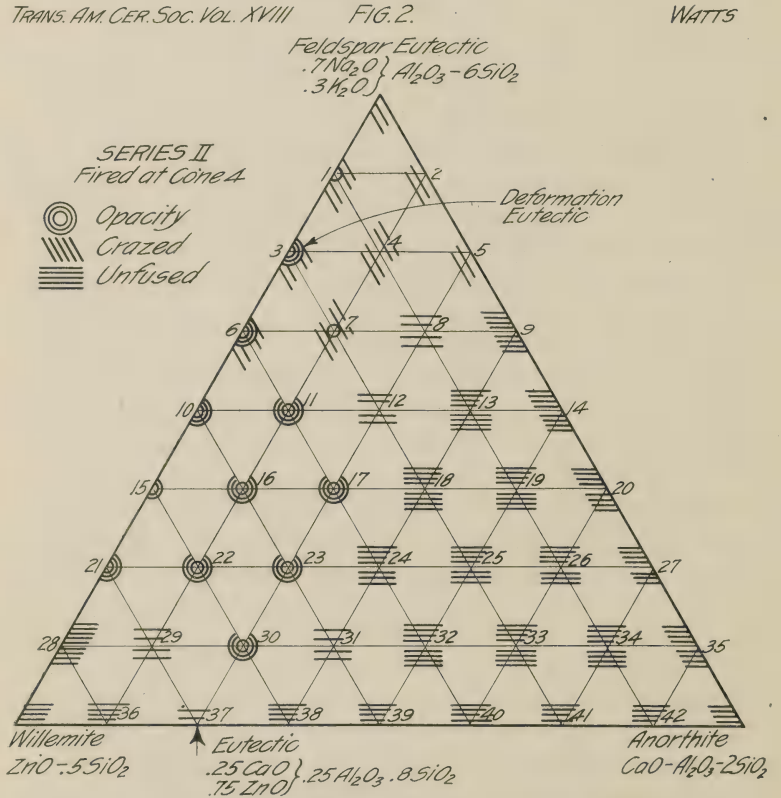
Opacity. The opacity of this series aside from that which is imparted by the semi-fused feldspar is confined to those members containing .25 equivalents or less CaO and .25 equivalents or more ZnO, and increases with decrease of CaO and increase of ZnO content.

Crazing. Crazing occurs in this series only when the feldspar content exceeds .5 equivalents and the CaO content is less than .25 equivalents.

Surface Texture. This entire series consists of mat glazes or only semi-bright glazes, and all the members containing anorthite norm resemble fused slips. Only one member, Series II, Member 22, has sufficiently fused to permit crystals to form, and these appear only when a temperature of cone 8 is reached.

General Conclusions. In this study the evidence obtained indicates that if the glaze is to be considered from its so-called norm content, the following conclusions may be drawn. 1. The temperature of maturity of a glaze is considerably lower if the

CaO is present as wollastonite norm ($CaO\text{-}SiO_2$) than if present as anorthite norm ($CaO\text{-}Al_2O_3\text{-}2\ SiO_2$). 2. The presence of anorthite norm tends to produce slips, while the presence of wollastonite norm tends to produce glasses. 3. The presence of willemite norm ($ZnO\text{-}.5\ SiO_2$) very materially lowers the



temperature of maturity of the glaze from that obtained by either feldspar-wollastonite or feldspar-anorthite mixtures. 4. A low feldspar content (less than .5 equivalents in anorthite bearing glazes and less than .375 equivalents in wollastonite bearing glazes) is necessary to insure against crazing and with this must be coupled more than .25 equivalents willemite norm if the glaze is a glassy one.

PRACTICAL HINTS ON THE BURNING OF SALT GLAZED VITRIFIED HOLLOW WARE

S. C. KARZEN

Defects in sewer pipe are quite numerous, and appear at the most unexpected times, but all the troubles can be traced back to some definite cause, and if each of these causes be eliminated, most assuredly the troubles will disappear.

The most common trouble encountered in burning is the so-called blowing or scaling. This is done during the water smoking period and as we are all aware by lengthening the period, this defect may be eliminated. Altho this knowledge is quite common among clay workers, it is really surprising how few of them know how to proceed when the trouble shows up.

It is for the unknowing ones that the following chart has been drawn up. It is self-explanatory, and can be used with any type of clay, and I am quite sure that if a little judgment is used along with the chart, good results may be obtained.

As may be noted in Table I, the burning period is divided into days, the length of the period depending on the size of the largest ware set. Ware manufactured of shale ordinarily takes longer periods of burning, due to excessive amount of carbon present. Fireclay ware requires shorter periods as noted. A series of cut and try methods must be used to determine the conditions of different plants. For example, we set a kiln of 18 in. D. S. pipe and mark the burn nine days as per Table I. The burning method as outlined on the chart for a nine-day kiln is then followed. We find that the ware is all right when drawn. The next kiln of ware (18 in. D. S.) we set, we mark up 8 days and follow the schedule for an 8-day kiln. When the kiln is drawn the ware is noted, and this method is followed until we see that if we cut the time any further trouble will result, and at this point it is wise to stop.

Using this method of burning, it is readily seen that a close tab can be kept on ignorant burners. Also placing burners on a time schedule for the preliminary heating up, brings kilns off on time. Relative to the amounts noted per firing, this was based on Pittsburgh and Ohio coals. The amounts should be slightly increased for western coals which are of a lower calorific value. Following the chart, with a little experimenting, it is a simple matter to eliminate blowing and scaling, with the least possible increase in burning time. One thing must be noted,however, in this regard, and that is, that ware set green is a fertile cause for blowing, and that in all cases where this is found necessary an extra day or perhaps more should be allowed in burning.

Fire Cracking. This is a defect quite prevalent in the sewer-pipe industries, and a large percentage of loss can be attributed to it. There are several reasons for this defect, any one of which may cause a considerable amount of loss. If the pipe are set green, the chances are that a number of them will be fire-cracked when drawn, especially, if the clay happens to be short naturally. Hence in a case of this kind, it is necessary that ware be bone dry when set.

Another common cause is in the setting. Large pipe are set 4 high or 8 feet in all. In setting it is necessary that the upper pipe be perfectly centered in the pipe beneath. Failure to do so means unequal distribution of the weight of the pipe on the socket of the pipe beneath, which results in a cracked pipe. Too much emphasis cannot be placed on the care to be used in setting ware.

The third cause, and the one least understood, is that due to too rapid heat raising, caused by too heavy firing at any one period, and too long periods between fires. This causes the ware to heat up rapidly during the combustion of the fuel, and expand at the same time. The period between fires causes cooling in the ware, consequent contraction, and eventual cracking. The most sensitive point is as a rule the socket, and this is where the most cracking occurs. In order to eliminate losses of this kind, it is first necessary to avoid too heavy firing. The cleaning of the fires is the greatest danger period. Instead of cleaning every fire hole around the kiln, every other hole should be cleaned, thus permitting half the holes to admit cooling oxygen during the clean.

Also short firing periods should be observed during the heat raising, and light fires used. This will be saving on the fuel, as well as reducing the fire cracking, but it will of course mean extra labor in firing. At one plant where it was almost impossible to burn large ware without excessive loss, the mere shortening of the firing period and the reduction of the amount used to a firing, almost eliminated this trouble.

Swelling. So much has been written on this subject and so fully has it been discussed, that I think it unnecessary to devote much space to this subject. Suffice to say that this trouble is directly due to improper oxidation of the carbon contents of the clay. Very little swelling is ever incurred in the fire clay districts, not because carbon is lacking in the clay, but because it is in smaller percentages, and in a form much more easily oxidized. Many fire-clay pipes show red centers which means too rapid oxidation, but swelling seldom occurs. Shale wares, however, show rapid oxidation by swelling, caused by the effort of the CO_2 to escape from the ware after partial vitrification has set in.

The one remedy is the use of large trials, which method has been amply described in numerous articles.

Scumming. This is another defect which has been amply discussed. It may be due either to the presence of soluble salts in the clay or to those introduced with the tempering water in the preparation of the clay. This latter item is often overlooked. I know of a case in which soluble salts were introduced with the water used for tempering. The pipe showed up badly, scummed, and poorly glazed, even with use of excess barium carbonate. After the source of water was changed, the pipes were completely different in appearance. If change of water does not benefit, the only other recourse is to the use of a compound of barium.

Roughness — Crazing. There are a number of defects which show up in the last stages of the burning and these will be discussed under one heading because the defects are all attributable to one cause.

Roughness is a defect common to vitrified ware in general. Much has been written on this subject and numerous theories

have been advanced, but roughness is just as common as it ever was. There are several varieties of roughness, or rather several causes to which roughness may be attributed, the first and last known is called iron spots. This is thought to be primarily caused by reducing action in the kiln, and will be discussed more fully later.

The second variety of roughness is due to the presence of carbon, generally in the form of a high carbon shale which does not become intimately mixed with the other clay in grinding. These particles remain in the surface of the ware and burn out eventually, leaving a cinder and very often a pitted surface. This is especially true in certain districts in Michigan, where a mixture of highly carboniferous shale and fire clay are used. The shale is so high in carbon that it burns out in spots, making a very rough looking pipe. In this particular case, the high carbon shale was eliminated from the mixture, and the roughness disappeared to a large extent.

Reduction During Salting Period and Its Effects. In the fire clay districts, where the clay naturally burns to a light buff color, it is the common custom to thoroughly smoke or reduce the surface of the ware in order to obtain the dark color demanded by the trade. In shale ware this is not necessary, as the material burns to a red color naturally. Moreover, reduction in shale gives the ware a highly metallic luster and black color which is very undesirable. But as I have said before, fire clay is being smoked to a large extent for the color. The result of this action is far-reaching on the quality of the ware, and has given rise to a number of defects which in many cases make the ware almost unmarketable. The first consequence of this custom is the fuel waste. In order to ensure reducing conditions, the burners lower the dampers, close the fires up tight, and fire at long intervals. This keeps the kiln full of smoke, which may be observed pouring from the stack at any time. This is naturally a very wasteful procedure from a fuel standpoint as the biggest percentage of the fuel added is being lost at the stack.

The second result I wish to call attention to, is not noticed until after the pipe have been exposed to the weather for awhile. After the atmospheric conditions have worked their will on the

stock pile for a few months, these pipe commence to craze, showing faint hair-like cracks all over the surface of the ware. This is never found in a pipe unless the surface has been subjected to severe reducing conditions. The action involved is probably a weakening of the body by the change of the iron from the "ferric" to the "ferrous" stage.

The third and by far the most important effect is the resulting roughness. Roughness is prevalent to a large extent in the fire clay districts, and to a less extent in the shale district. The roughness I am now referring to is that most commonly called ironing or iron spots. In spite of the fact that shale is supposedly not subject to reduction, roughness is quite common.

The usual explanation for ironing is based on the assumption that the reducing action of the gases of combustion brings these to the surface. In order to get at the truth of the matter, and determine just when this roughness occurs, a few simple experiments were made. Ware was set in different parts of a kiln in such a fashion that no glaze could penetrate. Also ware was set which was continually bathed by the salt fumes. When the ware was drawn it was found that the unglazed material was perfectly smooth and the glaze ware was quite rough. It was decided from this that roughness did not show up until the salting period. In order to verify this, an entire kiln of ware was heated up until ready to be salted and finished, and then allowed to cool without any salt being added. On opening the kiln, it was found that the ware was free from any iron spots. This same kiln was then closed again and rapidly fired and salted. When drawn, the ware was just as rough as usual. It left no reasonable doubt but that the iron spots are brought to the surface during the salting of the ware and not before. Hence we can reduce or oxidize as much as we please previous to this period but during salting oxidizing conditions must be obtained.

Color without Reduction: Since the trade demands colored ware, it behooves us to obtain it in the fire clay districts. However, as I have just pointed out, reduction must be avoided as much as possible especially during salting.

In this respect I wish to state that I have obtained splendid coloring without the aid of excessive smoking. During the heat

raising period it is often quite a problem to keep conditions oxidizing due to extremely heavy firing. Hence it is quite easy to obtain a thin coat of smoke in the ware at a single firing. This in turn can be easily drawn out again by opening of the fires for a short period. Then another skin of smoke can be allowed to penetrate the ware. This is again burned out by opening the fires. This is commonly called flashing in the fire-clay face-brick districts, and is their usual method for obtaining flashed shades. The alternate reducing and oxidizing leaves the iron on the surface in a reddish color. Now when the salt is applied, bright oxidizing fires may be used, and the cherry red coloration will be obtained.

Salting. I have stated that much of the roughness encountered in burning is due to reducing conditions set up during salting. The following scheme of salting was used with the primary object of creating oxidizing conditions. I have since found that exceptional glaze can be obtained in many cases where glazing was difficult before.

To begin with, the ware must be burned fairly hard before salting is considered. After the trials show up reasonably hard, the kiln fires must be prepared for salting as follows: — The fire holes must be thoroughly cleaned, the wisest plan being to clean alternate fire holes and thus eliminate excessive amounts of green fuel and the resulting heavy smoke. After the fires are clean, a light salt fire is placed close to the mouth of the fire hole, and a small air space is allowed over the top of the fire. This is in the case of dead bottom furnaces, but where horizontal bars are used it is best to leave the door open just a trifle. This may not be the most economical way of firing, but it ensures a sufficient supply of oxygen which is what is desired. The fire is allowed to burn until the green coal is aflame and the volatile matter is expelled. This ordinarily requires about 15 to 20 minutes with long flamed coals, and a little less with short flamed coals. At this stage the fires are at their greatest intensity. The salt is now applied, a small scoop should be used, and a small amount of salt spread evenly over the fire. Great care must be used that only a limited amount of salt be used to an application, for reasons which I will discuss. In the case

of dead bottom fire holes the furnace hole should be immediately closed up with a small scoopful of coal. With straight grates, all that is necessary is to close the door. A period of about ten minutes or so is now allowed until the salt decrepitates completely, and then another round is applied as stated, followed by a small amount of coal or the closing of the fire door. When the third round has been applied, and the salt burned, another light fire is put on. Allowing time for the volatile matter to be driven off, three more rounds are applied. Trials can be drawn every three rounds to determine the progress of the glazing period. The fires should be shaken or given a light clean after every three applications. Six rounds administered in this fashion, should be sufficient to develop a good glaze in most cases. Clays difficult to glaze may require three additional rounds applied after the method already outlined.

Considering the operation outlined step by step, we find that first the trial must be reasonably hard. This is well understood by most burners; but in the use of haphazard methods of salting, kilns are very often overburned, due to the burner waiting too long before starting the salt application. Using the method as outlined it is possible to start earlier in the game, before vitrification has even started. As the salting progresses, the ware continues to harden, since the salt fumes have a fluxing action. By drawing trials after every three rounds, it is possible to keep complete control of the salting; and when the trials finally show hard enough and glazed enough, salting may be discontinued. By thus starting the salting early, and hardening the ware gradually during the salting, it is possible to lessen the chances for over-burned kilns.

Cleaning of Fires: Most practical burners clean fires as a preliminary measure to the salting process, but they never go beyond that, and consequently lose the benefit of the preliminary work. By cleaning the fires before salting, we bring them to a high heat, and by keeping the fires bright and unclogged, we permit free oxygen to enter, thus furnishing good combustion conditions and making the kiln atmosphere oxidizing. The fires must be stirred up and kept bright continuously in order to ensure these conditions.

Salt Application: As stated 15 minutes must pass before salt is applied. This gives the fires an opportunity to attain their maximum temperature. Also the volatile matter of the coal has, to a large extent, been expelled and conditions in the kiln have become more oxidizing. Furthermore, on the application of each fire, there is always a slight scum of smoke on the surface of the ware which gathered while the volatile matter bathes the ware. This burns off immediately after the volatile matter is expelled, and the oxygen has a chance to get in its work. If the salt is applied before this scum is burned off, a rough product is ordinarily obtained, and in shale districts, a metallic black color results.

The salt is applied in small amounts and is well spread, because larger amounts will deaden the fires and remain unburned.

Coal is added in small amounts after each application of salt in order to keep the heat in the kiln to a maximum. The action of salt glazing is an endothermic one (heat absorbing), and it is necessary to add a little fresh fuel with each charge of salt in order to keep the kiln temperature uniform. A little fuel closes the mouth of the fire hole, where dead bottom grates are used, and cuts off the flow of cold air over the fire. Cold air should never be admitted over the fire except in cases where a kiln has extremely poor draft and conditions are very reducing. The admission of cold air over the fire bed has the effect of reducing its temperature, and thus cutting down its efficiency so far as decrepitating the salt is concerned. For best results, all air must be admitted through the fuel bed, and not over it.

The application of three rounds is not arbitrary. I have found by experiment, that three small rounds applied as stated is about as much as an ordinary salt fire can take care of efficiency. By the time a fourth round is to be applied, the fires have lost their brilliancy. On the other hand, if only two rounds are used, we do not get the most out of our fuel, and there is always an opportunity of over-burned ware due to the use of fuel to excess. After the third round has been applied, a small salt fire is used, which brings the fuel bed back to its original heat intensity.

Cleaning and stirring of the fires continually is necessary during the operation. It will be noted in this regard, that the salt to a certain extent melts and combines with the mineral matter of the coal ash to form a soft glassy slag which runs down through the fuel bed or grates as the case may be, hardens as soon as the cool air strikes it, and prevents the air from entering. Hence it is of vital importance that the bed be shaken, enough to break up this formation.

If the above method be followed and care taken, roughness can to a large extent be eliminated, and moreover an exceptionally bright salt glaze can be developed. Complete control of the glazing is possible at all times, and by the three round periods it is possible to salt until a good glaze is obtained.

As already stated, good color in the fire clay districts can be obtained without the excessive smoking, but the preliminary flash must be administered first.

Before leaving this subject I wish to state that at all times it is necessary to have a good draft during this operation. If a damper is used, it should be raised pretty well, thus taking the fumes rapidly through the kiln, and ensuring maximum oxidizing conditions.

Salt Scumming: It happens quite often that after a kiln is finished, and the ware drawn, that a white scum shows on the ware after it has been in the open a short time. This is due to excess salt fumes, in the kiln atmosphere, which had not been drawn off at the stack, but were redeposited on the ware. As a matter of course, the first heavy rain will remove this from the surface of the ware; but to the uninitiated, this often appears a serious defect, and may result in a loss of business. In order to eliminate this scum, a light fire should be applied after the salting has been completed, and most of the salt has decrepitated. At the same time the stack damper should be raised high. The added fire will have a tendency to burn up what salt may remain in the fire holes, while the increased draft will draw the fumes from the kiln. The small fire will not affect the ware, if it is not applied until the kiln has evidently started cooling. Scumming of this nature can be eliminated completely, in this way.

TABLE I. SHOVELS OF COAL EVERY 1½ HOURS

KIND OF WARE	LENGTH BURN IN DAYS		BURN	1ST DAY	2ND DAY	3RD DAY	4TH DAY	5TH DAY
	SHALE	FIRE CLAY						
			days	*shovels*	*shovels*	*shovels*	*shovels*	
8 in., 10 in., 12 in. dia...	5	4	4	2	2½	Kiln cleaned, heat raised	Finished	*
15 in. Single strength...	6	5	5	1½	2	2½	Fires cleaned, heat raised	Finish
15 in. Double strength..	7	5	6	1	1½	2	2½	Fires cleaned, and heat raised
18 in. Single strength...	8	5	7	1	1	1½	2	2½
18 in. Double strength..	9	6	8	½	1	1	1½	2
20 in. S. S.....	9	6	9	½	1½	1	1	1½
20 in. D. S.....	10	7	10	½ shovel † every other fire hole	½ all around	½	1	1

22 in. S. S.....	9	6	11	½ shovel every other fire hole	½ shovel every other fire hole	½ shovel, all holes burning	½	1
22 in. D. S.....	10	7	12	½ shovel every other fire hole	½ shovel every other fire hole	½ shovel, all holes burning	½	½

* NOTE — Coal fired whenever necessary after fires are cleaned.
† NOTE — Where marked ½ shovel every other fire hole is meant only ½ kiln is lighted up.

TABLE I (continued)

KIND OF WARE	6TH DAY	7TH DAY	8TH DAY	9TH DAY	10TH DAY	11TH DAY	12TH DAY
8 in., 10 in., 12 in. dia.	*shovels* *	*shovels*	*shovels*	*shovels*	*shovels*	*shovels*	*shovels* .....
15 in. S. S.	.....	.....	.....	.....	.....	.....	.....
15 in. D. S.	Finish	.....	.....	.....	.....	.....	.....
18 in. S. S.	Clean fires, raise heat	Finish	.....	.....	.....	.....	.....
18 in. D. S.	2½	Raise heat, clean fires	Finish	.....	.....	.....	.....
20 in. S. S.	2	2½	Clean fires, raise heat	Finish	.....	.....	.....

20 in. D. S.....	1½	2	2½	Clean fires, raise heat	Finish	.....	.....
22 in. S. S.....	1	1½	2	2½	Clean fires, raise heat	Finish	.....
22 in. D. S.....	1	1	1½	2	2½	Clean fires, raise heat	Finish

NOTE — Where marked "½ shovel every other fire hole," is meant only ½ kiln lighted up.

COMPARATIVE MEANS OF MAKING POROSITY AND ABSORPTION MEASUREMENTS

BY F. W. WALKER, JR.

During the past several years, I have been engaged in extensive researches requiring numerous porosity determinations of a fairly high degree of accuracy. The difficulties to be encountered in making these determinations were not fully realized at first, and as a result the value of much data now on file among my records is more or less limited.

When investigations are inextensive requiring but a few determinations, there is not apt to be any wide deviation in the results obtained. The specimens are made and treated under the same or very nearly the same conditions, and although the absorption of the specimen may or may not be complete, the values are fairly relative, regardless of the treatment used. But where investigations are more extensive, requiring the comparison of groups of trials made up and treated at different times and under varying conditions, these relative porosity determinations lose their comparative value, unless great care has been used to fully replace all free gases contained within the pores of the specimens.

The complete removal of the free gases contained within the pores of a specimen is not easily accomplished, and it is the purpose of this paper to throw some light upon the different means that have been used to procure absorption and porosity determinations.

There is little doubt in my mind but that such matters as structural density, size of pore, surface, and shape of the piece all have their effect upon the rate and amount of absorption, and especially where absorption is only partially complete. It is not the intention, herein, to treat this part of the question, for information of their character is difficultly obtainable, but from general observation and experience these factors are believed to be

worthy of consideration, and should not be overlooked in accounting for differences.

Absorption methods as reported, heretofore, in the *Transactions* can be divided into three general classifications:

1. Soaking in cold water.
2. Soaking in boiling water.
3. Soaking in water under vacuum.

Under these three main classifications, numerous methods can be had by considering the factor of time, which in the case of soaking in cold water varies from one to eight days and less time where boiling water or the vacuum is used.

Following an analysis of the various methods used, an investigation was outlined to determine the comparative merits of each, and the work so outlined as to roughly show the rate at which the air is removed by the different treatments. The methods used for obtaining absorption were as follows:

A—1. 5 hours soaking in cold water
2. 24 hours soaking in cold water
3. 48 hours soaking in cold water

B—1. 1 hour soaking in boiling water
2. 3 hours soaking in boiling water
3. 5 hours soaking in boiling water

C—1. 5 hours soaking in cold water under vacuum of approximately 28 inches
2. 24 hours soaking in cold water under vacuum of approximately 28 inches

All trials heated to boiling were cooled to room temperature by the addition of cold water before making their volume determinations.

The ten trials used for the work were biscuit wall tile, 6 in. by 3 in. by ⅜ in. which gave an average displacement volume of 116 cc., and in all tests were completely immersed standing on the long edge.

In order to secure a higher degree of accuracy and also to secure an idea of the amount of variation in check determinations, the work was carried out in duplicate using the same specimens.

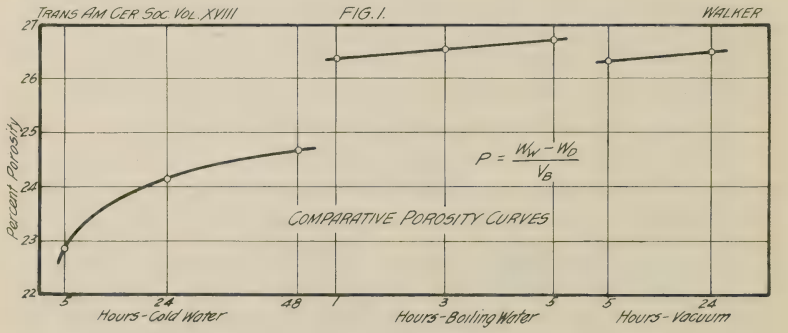
The results of this investigation are shown in Table I.

The formula used for computing these values is as follows:

$$P = \frac{Ww - Wd}{Vs} \times 100$$

where P = the percent porosity
Ww = the wet weight
Wd = the dry weight
Vs = the displacement volume of the saturated piece.

An analysis of these figures can be had best by referring to the curves in Figure 1, which shows that the soaking in boiling



Vacuum - in. Hg.	26.0	26.5	27.0	27.5	28.0	28.5	29.0	29.5
Boiling - Temp. F.	124	119	112	106	100	90	78	58
Vol. Reduction Percent	13.3	11.7	10.0	8.3	6.7	5.0	3.3	1.7

water for five hours gives the highest results and that very little absorption takes place after the first hours boiling. The cold water soaking proved the least efficient but showed little variation, while the vacuum system gave high results but showed considerable variation.

The failure of the vacuum method to give a maximum figure as well as a reliable figure, was other than expected, but an analysis of the rate of expansion of the air with variation of pressure, as shown by Table II, together with the difficulties encountered in maintaining a given vacuum without the aid of expensive equipment will largely account for the results.

Just what the action of the boiling water upon the trial piece is, is not clear, but the results seem to indicate that the steam bubbles have some ejecting or solvent action upon the gases.

Because of a lack of equipment, the boiling test under high vacuum of constant pressure was not made but judging from the results of these tests, together with the figures shown in Table II, this method should give the best results, providing a sufficiently high vacuum could be obtained to cause rapid boiling at room temperature. Where this test cannot be carried out, the five hour boiling test at atmosphere pressure, can be used with good results, but greater variation due to a change in the structure and shape of piece might be expected with this latter method.

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TABLE I. COMPARATIVE POROSITY VALUES

5 Hours Total Immersion — Cold Water.

Trial No.	No. 1	No. 2	No. 3	No. 4	No. 5	No. 6	No. 7	No. 8	No. 9	No. 10	Average
Test No. 1	22.44	22.13	22.27	22.40	23.16	23.03	23.10	22.69	22.94	23.14	22.73
Test No. 2	22.21	21.92	22.50	22.67	23.28	23.55	23.36	22.88	23.26	23.94	22.96
Difference	.23	.21	.23	.27	.12	.52	.26	.19	.32	.80	.....

Combined Average 22.845

24 Hours Total Immersion — Cold Water.

Trial No.	No. 1	No. 2	No. 3	No. 4	No. 5	No. 6	No. 7	No. 8	No. 9	No. 10	Average
Test No. 1	23.66	23.34	23.55	23.68	24.44	24.61	24.54	24.08	24.38	25.00	24.13
Test No. 2	23.39	23.35	23.49	23.54	24.37	24.61	24.64	24.04	24.46	25.15	24.10
Difference	.27	.01	.06	.14	.07	.00	.10	.04	.08	.15	.....

Combined Average 24.115

48 Hours Total Immersion — Cold Water.

Trial No.	No. 1	No. 2	No. 3	No. 4	No. 5	No. 6	No. 7	No. 8	No. 9	No. 10	Average
Test No. 1	24.11	23.70	24.22	24.17	24.96	25.36	25.16	24.70	25.01	25.41	24.68
Test No. 2	24.38	23.91	23.88	23.96	24.89	24.82	24.84	24.78	25.12	25.89	24.65
Difference	.27	.21	.34	.21	.07	.54	.32	.08	.11	.48	.....

Combined Average 24.665

1 Hour Total Immersion — Boiling Water.

Trial No.	No. 1	No. 2	No. 3	No. 4	No. 5	No. 6	No. 7	No. 8	No. 9	No. 10	Average
Test No. 1	24.89	25.49	25.66	25.94	26.75	26.94	26.88	26.25	26.50	27.18	26.25
Test No. 2	25.97	25.64	25.82	25.93	26.82	27.02	26.95	26.38	26.68	27.43	26.46
Difference	1.08	.15	.16	.01	.07	.08	.07	.13	.18	.25	.....

Combined Average 26.355

3 Hours Total Immersion — Boiling Water.

Trial No.	No. 1	No. 2	No. 3	No. 4	No. 5	No. 6	No. 7	No. 8	No. 9	No. 10	Average
Test No. 1	25.76	25.39	25.58	25.90	26.70	27.07	26.88	26.37	26.66	27.35	26.37
Test No. 2	26.15	25.73	25.98	26.16	27.05	27.46	27.12	26.61	26.88	27.52	26.67
Difference	.39	.34	.40	.26	.35	.39	.24	.24	.22	.17	.....

Combined Average 26.52

5 Hours Total Immersion — Boiling Water.

Trial No.	No. 1	No. 2	No. 3	No. 4	No. 5	No. 6	No. 7	No. 8	No. 9	No. 10	Average
Test No. 1	26.10	25.76	25.97	26.19	27.02	27.25	27.18	26.58	26.91	27.56	26.65
Test No. 2	26.07	25.72	26.04	26.25	27.11	27.34	27.40	26.80	26.98	27.58	26.73
Difference	.03	.04	.07	.06	.09	.09	.22	.22	.07	.02	.....

Combined Average 26.69

5 Hours Total Immersion — 28 inches Vacuum.

Trial No.	No. 1	No. 2	No. 3	No. 4	No. 5	No. 6	No. 7	No. 8	No. 9	No. 10	Average
Test No. 1	25.55	25.47	25.57	25.55	26.62	26.90	26.69	26.18	26.54	27.25	26.23
Test No. 2	25.88	25.55	25.65	25.87	26.88	26.91	26.85	26.25	26.55	27.26	26.37
Difference	.33	.08	.08	.32	.26	.01	.16	.07	.01	.01	.....

Combined Average 26.30

24 Hours Total Immersion — 28 inches Vacuum.

Trial No.	No. 1	No. 2	No. 3	No. 4	No. 5	No. 6	No. 7	No. 8	No. 9	No. 10	Average
Test No. 1	25.75	25.39	25.54	25.74	26.65	26.80	26.71	26.18	26.54	27.22	26.25
Test No. 2	26.22	25.81	25.97	26.15	27.06	27.19	27.12	26.60	26.92	27.64	26.67
Difference	.47	.42	.43	.41	.41	.39	.41	.42	.38	.42	.....

Combined Average 26.46

CLAY AND SHALE RESOURCES IN THE VICINITY OF CLEVELAND, OHIO

BY FRANK R. VAN HORN

The clay industry in Cleveland and vicinity includes the manufacture of common brick, vitrified brick, hollow ware, and a small amount of drain tile. The region has a daily capacity of approximately one million standard brick. If this amount were produced during ten months of the year at an average price of six dollars a thousand, the annual value of the industry would amount to about one and one-half million dollars. This estimate is probably conservative as there are few firms that do not work during the entire year. Furthermore, a large amount of paving block which sells at from 13 to 18 dollars a thousand, is produced. Since Ohio, for some years, has ranked first in the United States with a production of 30,000,000 dollars worth of clay products, it can be seen that the Cleveland area is not an unimportant factor.

Clay. The clays of the region are chiefly transported clays of glacial origin. A few, however, are probably of lacustrine origin. They are either yellow or blue in color and vary in thickness from 3 to 75 feet. However, in one place the thickness was showed by boring to be over 150 feet. The blue clays are very dense and tough, and dynamite is sometimes employed in the mining operation. A variable amount of boulders is generally found, and in some places these consist of granites, gneisses, schists, and of several different kinds of limestone that are a source of trouble since small pieces may get into a brick. On burning, these particles become converted into lime which absorb moisture, and cause the brick to chip off or even break into several pieces. There are some alluvial clays along the rivers and streams, but none are used for commercial purposes. The nearest approach to a residual clay is what has been called the De Kalb[1]

[1] J. E. Lapham and Charles M. Mooney, *Soil Survey of the Cleveland Area, Ohio. Bull. of Bureau of Soils,* U. S. Department of Agriculture, Washington, 1906, p. 19.

clay. This is found in a narrow strip which is one-quarter of a mile to a mile in width and goes parallel to Lake Erie from Dover Bay through Lakewood and Nottingham toward Wickliffe. The De Kalb clay has been formed from the weathering of the Chagrin shale, although probably assisted by wave action of Lake Erie when at a higher level. An ultimate chemical analysis of this clay, and also one of the common blue clays formerly called by Newberry, Erie clay, follows:

Analyses of Clays, Cleveland, Ohio

	1	2
Silica (SiO_2)	62.25	59.70
Alumina (Al_2O_3)	20.35	14.80
Iron oxide (Fe_2O_3)	5.30	4.60
Magnesia (MgO)	1.01	5.14
Lime (CaO)	0.60	8.90
Soda (Na_2O) + Potash (K_2O)	2.46	3.40
Combined water (H_2O)	7.25	4.00
Sulphur trioxide (SO_3)	0.34	———
	99.66	100.54

1. De Kalb Clay (residual) from John Kline Brick Co., Wickliffe, Ohio. Chemists, F. J. Peck & Co.

2. Erie Clay, a blue drift clay used by some of the brick makers of this vicinity, Ohio Geol. Survey, Vol. 1, 1873, Page 177, Chemist, Dr. T. G. Wormley.

If analysis 1 is compared with that of the Chagrin shale, No. 3, a great similarity will be seen. The De Kalb clay shows a slight loss of soluble constituents, such as alkalies, lime, magnesia, and iron. This clay, Figure 3, is used only incidentally as it falls into the pit of Chagrin shale which is employed at this place for the manufacture of vitrified brick. If, however, the clay were present in sufficient amounts, it could be used in making brick. The sulphur shown in the analysis comes from undecomposed pyrite concretions which are present in the shale. Analysis

2 shows a large percentage of lime and magnesia which probably originated from the various kinds of limestone pebbles that generally occur in the drift.

When the field work on this paper was completed in the fall of 1909, there were in the district thirteen firms which employed clay in the manufacture of common brick. The daily production was about 600,000 standard brick, but the probable capacity was nearly 800,000. Two firms make a small amount of drain tile as well as common brick. The names of these firms, their location, and details about their clays are given in Table I.

TABLE I. CLAY BRICK MANUFACTURERS, CLEVELAND, OHIO

NAME OF FIRM	LOCATION OF PLANT	KIND AND THICKNESS OF CLAY USED
1. Barkwill Brick Co.	Foot of East 75 St. in Kingsbury Run.....	10 to 25 feet of yellow clay with variable amount of glacial boulders.
2. Brookside Brick Co.	West 56th St., near Dennison Avenue ...	Mostly yellow clay with variable amount of boulders. When visited, they were working on 4 feet of yellow clay, and 14 feet of tough blue clay.
3. Cullen Brothers.	Foot of Reeves Ave. S. E. near corner of Independence Road and entrance to Forest City Park.	12 feet of yellow clay with few pebbles, and 8 feet of tough blue clay quite free from pebbles.
4. Duty and Company.	Foot of East 77th St. in Kingsbury Run.	10 to 25 feet of yellow clay with variable amount of pebbles.
5. Farr Brick Co.	Foot of Reeves Ave. S. E. near corner of Independence Road and entrance to Forest City Park.	15 feet of yellow clay with practically no pebbles.

TABLE I — Concluded

NAME OF FIRM	LOCATION OF PLANT	KIND AND THICKNESS OF CLAY USED
6. G. H. Gynn.	Harwood Ave., and East Beyerle St. S. E., near entrance to Forest City Park.	6 to 8 feet of yellow clay underlain by 10 to 15 feet of blue clay, with hardly any pebbles or boulders.
7. W. H. Harrison.	Near corner of Guy and East 49th St., S. E.	10 to 12 feet of jointed dense blue clay capped and underlain by 2 feet of stratified clay. Considerable amount of pebbles.
8. Standard Brick Co.	Bradley Road and Baltimore and Ohio R. R., South Brooklyn.	50 to 75 feet of dense blue clay with variable amount of boulders. Borings show 80 to 90 feet more clay.
9. Wilhelian Thies.	Ridge Road near Cleveland, Lorain & Wheeling R. R., and West Park Cemetery.	7 feet of yellow clay with few boulders.
10. Towle Brick Co.	Bradley Road and Baltimore & Ohio R. R., South Brooklyn.	20 feet of yellow clay underlain by 30 feet of tough blue clay, variable amount of pebbles.
11. John Wilson.	Foot of Independence Road on Ohio Canal opposite Cleveland Furnace Co.	10 to 12 feet of blue clay with small amount of pebbles.

DRAIN TILE AND CLAY BRICK PRODUCERS

1. John Perram & Son	Independence, Ohio.	3 feet of blue clay.
2. J W Spencer & Son.	Rocky River, Ohio. R. F. D. No. 64, near White's Villa on Cleveland and Elyria Trolley Line.	3 to 5 feet of light grayish-blue clay. It is probably Dunkirk clay.

The eleven clay brick manufacturers use the "soft-mud" or "sand-mold" process in which the following stages may be distinguished: (1) mining and hauling the clay; (2) crushing; (3) tempering; (4) sanding; (5) molding; (6) drying; (7) burning.

The clay is dug in open cuts by steam shovel or by hand, and hauled in cars, carts, or wheelbarrows to the crushing rolls. The finely crushed clay is then tempered or mixed in a pug mill with water to give it the required degree of plasticity. The pug mill passes the plastic clay into the brick machine, or press, which forces the soft brick into previously sanded molds. The molds are emptied on wooden or steel pallets which are placed upon racks where the bricks are dried from nine hours to seven days, depending upon the kind of drying system in use. The air shrinkage of a brick made from one of the yellow glacial clays was determined. A green brick measuring 4⅛ by 9⅛ by 2⅜ inches, or 89.39 cubic inches, and weighed $6^{9}/_{16}$ pounds. A dried brick ready for the kiln measured 4 by 8⅜ by 2⅜ inches, or 81.93 cubic inches, and weighed 5½ pounds. This shows an air shrinkage of 7.46 cubic inches or 8.34 percent and a weight loss of $1\ ^{1}/_{16}$ pounds, or 16.19 percent. This loss shows that 15 tons of water must be evaporated if 30,000 bricks are made daily. After drying, the bricks are burned in rectangular up-draft kilns which with one or two exceptions, belong to the type known as the Dutch or Pennsylvania kiln. The fuel in most cases is natural gas, which is supplied from the West Virginia and Cleveland fields. It is said to cost in the long run about the same as coal, but is much cleaner and more easily regulated. The bricks are burned from five to eight days, and

FIG. 1

the temperature varies from 1500 degrees to 1600 degrees F. In order to determine the fire shrinkage, eight bricks were selected from different yards:

TABLE II

INTENSITY OF BURNING	DIMENSIONS IN INCHES	VOLUME IN CUBIC INCHES	WEIGHT IN POUNDS
Very hard burned	3⅜ by 8¼ by 2¼	62.64	4½
	3½ by 8⅛ by 2¼	63.98	4½
Hard burned	3¾ by 8½ by 2¼	71.70	4½
	3⅝ by 8 by 2¼	65.25	$4^{6}/_{16}$
	3¾ by 8¼ by 2⅜	73.48	4½
Soft burned	3⅞ by 8½ by 2¼	74.09	$4^{2}/_{16}$
	3¾ by 8½ by 2⅜	75.70	4½
	3⅞ by 8½ by 2⅜	78.17	$4^{11}/_{16}$

The volume of the three types of brick selected shows very clearly the intensity of burning as the fire shrinkage becomes greater with increasing heat. The average volume is 70.62 cubic inches whereas that of a dried brick was 81.93 cubic inches. This gives a fire shrinkage of 11.31 cubic inches or 13.8 percent. The air shrinkage was 8.34 percent. The average weight of the burned brick was $4^{7}/_{16}$ pounds, and that of the dried brick was 5½ pounds. This gave on burning, a loss of $1^{1}/_{16}$ pounds or 19.3 per cent. Compared with a green brick, it was found that a soft-mud brick in changing to the finished product lost 18.77 cubic inches or 20.99 percent and that the total loss in weight was $2^{2}/_{16}$ pounds or 32.38 percent.

The two drain-tile producers of the district use the stiff-mud process in the manufacture of drain tile and clay brick. Their methods of crushing and drying are the same as those of the soft-mud brick makers, but the molding and burning processes are those of the shale brick manufacturers. The sizes of drain tile most used are 12 inches long and 2½ to 3 inches in

FIG. 2

diameter. A great many are used in the district because of the pronounced clayey character of the soils.

Shale. The shales of the region that are used for economic purposes are the Chagrin of the uppermost Devonian, and the Cleveland, Bedford, and Orangeville of the Mississippian. The Chagrin, Figures 1, 3, 4, 5, is prevailingly of grayish blue color, while the Cleveland, Figures 1, 4, 5, is generally black with some layers of dark blue. The Bedford is both blue and red, and the Orangeville, Figure 2, is black. Pyrite is present in most of the shales, and in some horizons of the Chagrin, clay ironstone concretions are very prominent. Several ultimate chemical analyses of the various shales were collected from some of the brick companies and are given in Table III. The analyses tabulated are of clays listed as follows:

SHALES OF TABLE III.

1. Chagrin shale, blue, Cleveland Brick and Clay Co., F. J. Peck and Co., chemists.
2. Chagrin shale, blue, Cleveland Brick and Clay Co., F. J. Peck and Co., chemists.
3. Chagrin shale, blue, John Kline Brick Co., F. J. Peck and Co., chemists.
4. Chagrin shale, brown, Newburg Brick and Clay Co., F. J. Peck and Co., chemists.
5. Chagrin shale, blue, Newburg Brick and Clay Co., F. J. Peck and Co., chemists.
6. Chagrin shale, blue, Newburg Brick and Clay Co., F. J. Peck and Co., chemists.
7. Chagrin shale, blue, Ohio Clay Co., Oscar Textor, chemist.
8. Cleveland shale, black, Cleveland Brick and Clay Co., Oscar Textor, chemist.
9. Cleveland shale, black, Cleveland Brick and Clay Co., F. J. Peck and Co., chemists.
10. Cleveland shale, black, Cleveland Brick and Clay Co., F. J. Peck and Co., chemists.
11. Cleveland shale, black, Brower Farm, Ohio Clay Co., Oscar Textor, chemist.
12. Cleveland shale, black, Brower Farm, Ohio Clay Co., Oscar Textor, chemist.
13. Cleveland shale, black, Newburg Brick and Clay Co., F. J. Peck and Co., chemists.

TABLE III. ANALYSES OF SHALES FROM THE VICINITY OF CLEVELAND, OHIO

	Chagrin Shale							Cleveland Shale		
	1	2	3	4	5	6	7	8	9	10
Silica (SiO_2)	58.71	56.40	57.80	61.20	61.90	62.60	59.35	55.13	60.26	59.85
Alumina (Al_2O_3)	21.90	23.02	21.11	19.21	21.55	21.10	20.05	19.99	20.57	18.60
Iron oxide (Fe_2O_3)	4.55	4.50	5.80	4.55	4.25	4.40	8.05	5.28	4.00	4.50
Magnesia (MgO)	2.00	1.64	1.73	1.46	1.44	1.40	1.51	1.33	1.85	1.40
Lime (CaO)	1.67	0.85	0.80	2.00	1.50	1.54	1.20	0.44	0.48	0.40
Soda (NaO) / Potash (K_2O)	.....	.....	2.95	.....	.....	.....	4.11	3.37	.....	.....
Combined water (H_2O)	6.64	5.91	6.01	9.13	4.96	4.12	5.30	6.66	3.34	6.23
Sulphur trioxide (SO_3)	2.57	0.86	1.22	0.43	0.20	1.05	.....	.....	3.52	2.05
Carbon (C)	0.00	0.00	0.00	0.00	0.00	0.00	0.40	7.14	4.47	3.93
	98.04	93.18	97.42	97.98	95.80	96.21	99.97	99.34	98.49	96.96

TABLE III — Concluded

	Cleveland Shale						Bedford Shale		
	11	12	13	14	15	16	17	18	19
Silica (SiO_2)	56.10	58.10	55.10	59.90	62.77	58.60	57.28	57.40	56 61
Alumina (Al_2O_3)	17.18	15.50	20.86	26.28	24.93	19.50	21.13	21.20	21.63
Iron oxide (Fe_2O)	4.42	4.22	4.30	4.35	4.55	6.05	8.52	6.57	7.08
Magnesia (MgO)	1.24	1.08	1.46	1.84	1.40	1.11	2.13	1.40	1.41
Lime (CaO)	0.15	0.05	1.80	0.60	0.92	1.10	5.79	1.00	1.11
Soda (Na_2O)	.....	2.91	.....	.....	.....	3.36	.....	4.10	3.51
Potash (K_2O)	2.67							1.00	0.48
Combined water (H_2O)	6.49	7.02	7.30	5.40	4.35	7.08	5.22	7.75	6.53
Moisture	.....	.....	.....	.....	.....	.....	.....	.....	2.00
Sulphur trioxide (SO_3)	.....	.....	3.13	1.67	0.96	.....	.....	.....	.....
Carbon (C)	11.01	10.50	5.85	0.32	0.00	3.28	.....	.....	.....
	99.26	99.36	99.87	100.86	99.88	100.08	100.07	100.42	100.36

14. Green repressed paving block made by Cleveland Brick and Clay Co., consisting of Chagrin and Cleveland shale with very little glacial drift. F. J. Peck and Co., chemists.
15. Green repressed paving block made by Newburg Brick and Clay Co., composed of Chagrin shale with a little Cleveland, but more glacial drift. F. J. Peck and Co., chemists.
16. Average of seven samples taken in proportion to the thickness of strata from the side of a cliff consisting of Chagrin and Cleveland shale with glacial drift, made for the Ohio Clay Co., Oscar Textor, chemist.
17. Bedford shale, Northern Ohio, presumedly from South Park, or Independence, Cuyahoga Co., *Ohio Geol. Sur.,* Vol 7, pages 133-134, chemist not given.
18. Bedford shale, Northern Ohio, same as 17, but different sample and chemist.
19. Average of 10 samples of shales chiefly of Carboniferous age, used for making vitrified brick, block, and sewer pipe elsewhere than in the Cleveland district. *Ohio Geol. Sur.* Vol. 7, pages 133-134.

These analyses were commercial ones in which the determination of certain substances only was asked, which accounts for the fact that several substances were not determined and that very few analyses have a total of 100 percent. In analysis 3, the water was determined by ignition with a deduction for the sulphur, while in 7, 8, 11, 12, and 16, a similar calculation was made with the carbon. A comparison of the various analyses will show a great similarity in the composition of the three shales used for making brick, although they are of different geological age. Comparing the Chagrin with the Cleveland, the silica, alumina, magnesia, and lime have a tendency to be slightly higher in the Chagrin. The Cleveland is always immediately recognized by the amount of carbon present. The percentage of iron is higher in the Bedford, as is indicated by its generally red color. The constant amount of sulphur trioxide (SO_3) is due to the presence of pyrite (FeS_2). In analyses 7, 8, 11, 12, and 16, sulphur was certainly present and has been included under water as ignition. Analysis 19 shows that the shales of the district conform closely to the composition of other shales used in Ohio for the same purpose. Analyses 14 and 15 show the exact composition of the mixture used in a brick. Judging by the amount of carbon present, Analysis 14 shows that less than 20 percent

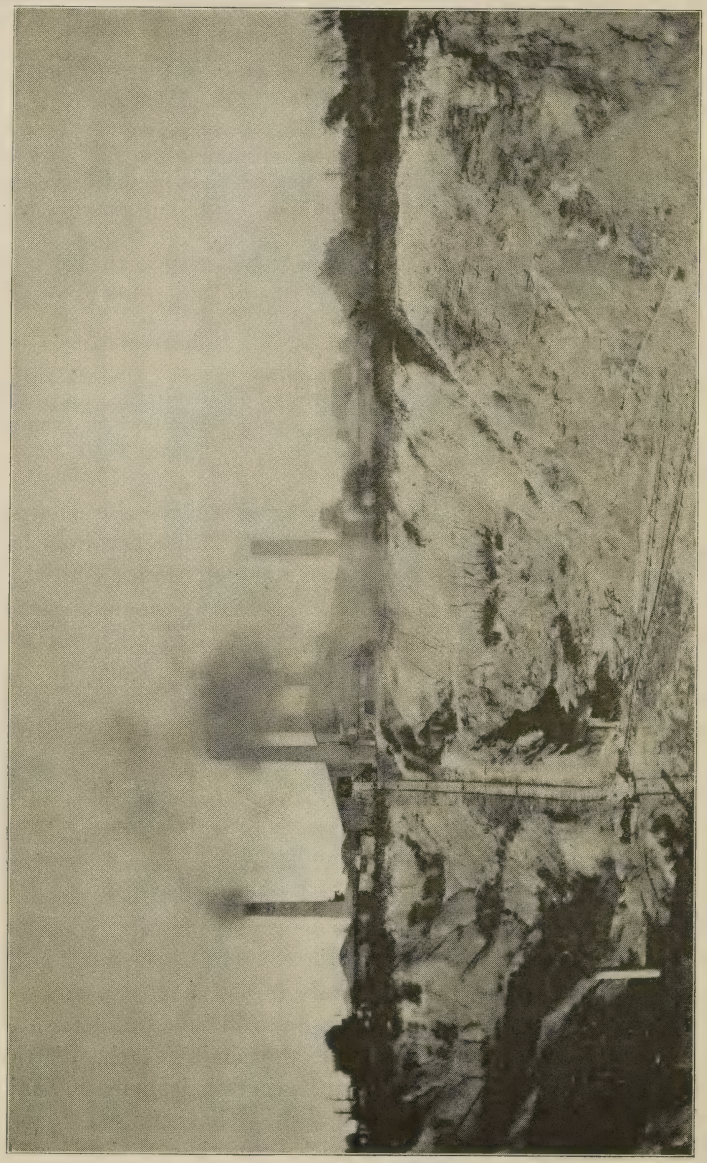


FIG. 3

of Cleveland shale must have been employed. In the shale bank of this company, Figure 1, there are three feet of glacial drift followed by two to three feet of blue Bedford. Under this, follow 18 to 20 feet of Cleveland and 88 feet of Chagrin shale. The section, Figure 5, used for making the brick analysed in 15, consists of 16 feet of drift clay, 6 feet of Cleveland and 113 feet of Chagrin. The glacial clay was undoubtedly richer in silica due to the presence of some quartz sand, which raised the percentage of silica higher than in any of the shales. On the other hand, the six feet of Cleveland was not sufficient to make any impression on the analysis if judged by the carbon content. There are some authorities who have been disposed to assert that the Cleveland shale could not be used for brick making. As a matter of fact it is said, by at least one brick manufacturer, that the Cleveland makes better brick than the other shales. However, it must be burned more slowly, and for a longer period in order to drive off all the carbon before any vitrification takes place. Otherwise, the various gases would be shut up in the interior of the brick, causing it to warp and bloat. A kiln of brick consisting entirely of Cleveland shale was made on January 19 and 20, 1904, by the Cleveland Brick and Clay Co. The kiln was packed with 19,800 standard brick, and 28,500 five inch paving block. The result after burning was 19,800 standards, all good; 26,632 No. 1, five inch; 1,440 No. 2, five inch; and 432 culls. This was claimed to be a better result than was usually obtained from the mixture composed chiefly of Chagrin, with very little Cleveland.

In 1909, when the field data were collected, there were 10 companies using shales for various purposes. Of these, eight were engaged in the manufacture of vitrified brick and paving block with a daily output of nearly 300,000 standard brick, or approximately 175,000 paving block of the largest size (five inch). Two firms produce only hollow ware, such as hollow brick, fireproofing, and conduits. Table IV shows the location of these companies with the kind and thickness of shale used:

TABLE IV. SHALE BRICK PRODUCERS NEAR CLEVELAND, OHIO

NAME OF COMPANY	LOCATION OF PLANT	KIND AND THICKNESS OF MATERIAL USED
1. Cleveland Brick and Clay Co.	Foot of Rosewood St. off Warner Road or Baltimore and Ohio R. R. south of Harvard Road, Mill Creek valley.	3 feet of yellow drift clay, 2 feet of blue Bedford, 20 feet of black and blue Cleveland, 88 feet of Chagrin.
2. Cleveland Hydraulic Press Brick Co.	South Park Station, Baltimore and Ohio R. R.	30 feet of red Bedford, 20 feet of blue Bedford.
3. Deckman-Duty Brick Co.	Corner of Adams Ave. and Sheffield St., Collinwood.	70 feet of Chagrin.
4. Forest City Brick Brick Co.	West 56th St. and Cleveland, Lorain and Wheeling R. R.	50 feet of Chagrin.
5. John Kline Brick Co.	Wickliffe, Lake Co., Stop 21, Cleveland, Painesville and Eastern R. R.	A few inches of De Kalb clay and about 75 feet of Chagrin.
6. Lakewood Brick Co.	S. E. corner of Hilliard Road and Elmwood Ave., Lakewood.	2 feet of yellow clays, 5 feet black Cleveland,
7. Metropolitan Paving and Brick Co.	1 mile E. S. E. of Willow Station, Baltimore and Ohio R. R. near Canal Road.	30 to 60 feet of Orangeville, a small amount of Chagrin which occurs near the plant is mixed with the Orangeville.
8. Newburg Brick and Clay Co.	One-half mile east of junction of Warner and Canal Roads.	16 feet of yellow glacial clay, 6 feet of black Cleveland, 113 feet of Chagrin.

MANUFACTURERS OF HOLLOW WARE

NAME OF COMPANY	LOCATION OF PLANT	KIND AND THICKNESS OF MATERIAL USED
1. Camp Conduit Co.	1½ miles S. W. of Willow Station, Baltimore and Ohio R.R., near junction of West Road and Rockside Avenue.	8 feet of red Bedford.
2. Ohio Clay Co.	One-half mile east of junction of Warner and Canal Roads.	5 to 10 feet of yellow drift clay, 128 feet of Chagrin but only 50 feet were mined.

Shale brick, paving block, and hollow ware are all made by what is called the stiff-mud process which differs from the soft-mud method in using less water for tempering. On this account the clay is less plastic, but the green product is easier to handle. This process requires more expensive machinery, and a larger force of men to operate it. The following stages may be distinguished in this method of manufacture: (1) Mining and transportation of the shale; (2) Crushing and screening; (3) Tempering; (4) Pressing or molding; (5) Cutting; (6) Repressing; (7) Drying; (8) Burning.

The shale is commonly obtained from the side of a valley, but two companies mine it from open pits which are about 75 feet deep, from which the drainage water must be continually pumped out. The unweathered shales are very dense and quite hard, so that all companies employ dynamite in breaking them up. A landslide[1] occurred on August 17, 1908, at the shale bank of the Cleveland Brick and Clay Co., Figure 1, which loosened from 60,000 to 80,000 tons and probably saved the company over $1,000 worth of dynamite. The method of transportation varies from a wheelbarrow to a small locomotive which is capable of drawing about five cars. The shale is crushed in dry pans, screened, and tempered in pug mills which are longer

[1] Landslide Accompanied by Buckling, and Its Relation to Local Anticlinal Folds, by Frank R. Van Horn, Bull. Geol. Soc. of Am., Vol. 20, p. 625.

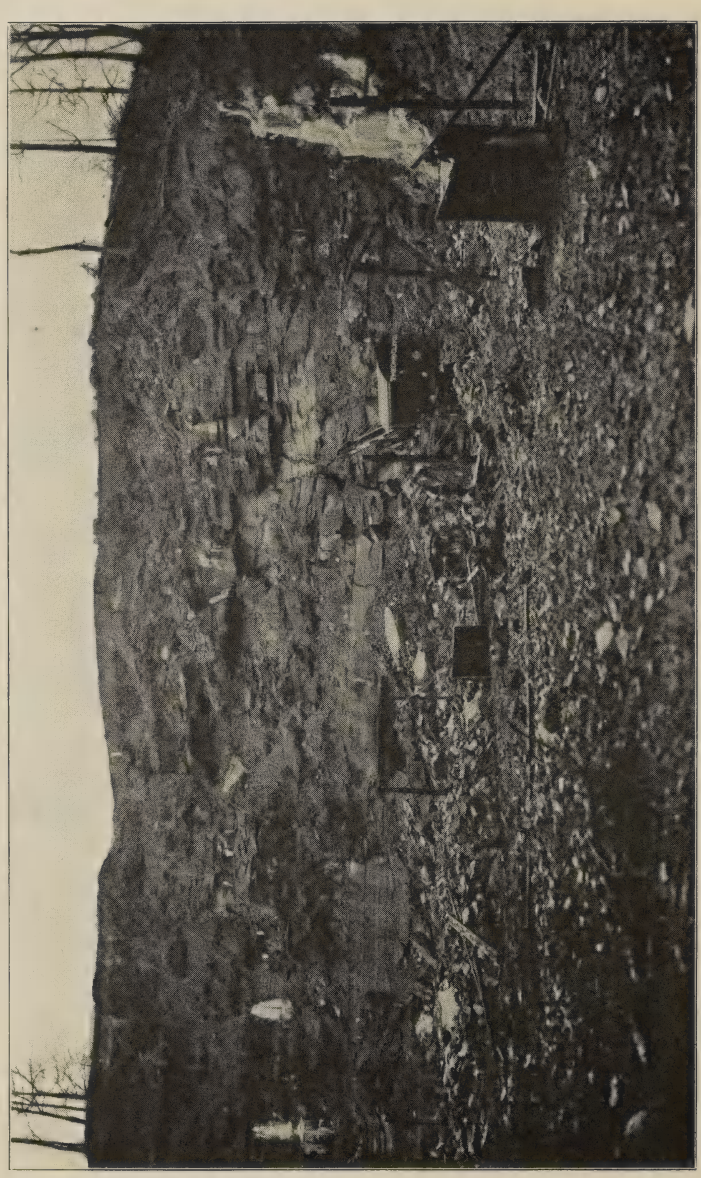


FIG. 4

than those employed in the soft-mud process since less water is used, and the shale requires more thorough mixing. The molding is done by the brick press which in all cases belongs to the type known as an auger machine. The shale is fed by pug mill directly to the auger machine, and issues from the die in the shape of a plastic bar which is cut by wires into the proper thickness since the die head gives the other dimensions correctly. Most firms cut standard brick and four inch paving block at the same time, but five inch paving block must be made separately. Some standard brick, and all paving block, go through a repress machine which makes the clay more dense, rounds off the corners, and allows the trade-mark to be stamped on. Some four inch block were found to decrease in volume about 6 percent, while five inch block lost about 16 percent by repressing. The brick are piled on cars and dried in tunnels, which in most places are heated by waste heat from the cooling kilns. The following data show the decrease in volume and weight of dried shale bricks when compared with green ones:

	inches	*cu. inches*	*pounds*
Standard brick, green	$4\frac{1}{2}$ by $9\frac{1}{2}$ by $2\frac{3}{4}$....	117.56	9
Standard brick, dried	$4\frac{3}{8}$ by $9\frac{1}{4}$ by $2\frac{3}{4}$...	111.28	$7\ \frac{13}{16}$
	Loss	6.28 or 5.34 percent.	$1\frac{3}{16}$ or 12.98 percent.
		cu. inches	*pounds*
4 inch paving block, repressed green,	$4\frac{5}{8}$ by $9\frac{7}{8}$ by $3\frac{5}{8}$.....	165.56	$12\ \frac{12}{16}$
4 inch paving block, repressed, dried,	$4\frac{3}{4}$ by $9\frac{5}{8}$ by $3\frac{5}{8}$.....	152.60	$11\ \frac{7}{16}$
	Loss	12.96 or 7.82 percent.	$1\frac{6}{16}$ or 13.46 percent.
		cu. inches	*pounds*
4 inch from another factory, green,	$4\frac{5}{8}$ by $9\frac{1}{2}$ by $3\frac{7}{8}$.....	170.25	$12\ \frac{12}{16}$
....., dried,	$4\frac{1}{2}$ by $9\frac{1}{4}$ by $3\frac{3}{4}$.....	166.09	$10\ \frac{9}{16}$
	Loss	14.16 or 8.31 percent.	$2\frac{3}{16}$ or 17 percent.

Average loss in volume of two 4 inch block was 8.06 percent, and the average loss in weight was 13.72 percent.

5 inch paving block,		
repressed, green, $5\frac{5}{8}$ by $9\frac{3}{8}$ by $3\frac{3}{4}$.....	197.75	15
repressed, dried, $5\frac{1}{2}$ by $9\frac{1}{8}$ by $3\frac{5}{8}$.....	181.92	$13\frac{9}{16}$
Loss	15.83 or 8 percent.	$1\frac{1}{16}$ or 9.58 percent.

The results show that the loss in volume is much less than the percentage of loss in weight. The standard brick and five inch block were from two other factories. When comparted with clay bricks under the same conditions, shale bricks show about the same decrease in volume, but a slightly smaller percentage of loss in weight in air shrinkage. The burning operation is entirely different from that of clay brick since all shale products are vitrified or partly fused, and the heat must be under better control. The kilns are all down draft ones, generally round, but in some places rectangular in shape. The maximum temperature used in these kilns was found to be from 1700 degrees to 1750 degrees F., determined by means of a pyrometer in one place, and by the use of Orton's fusion cone 07 to be 1850° F. in another locality. The fuel used in most places is soft coal. The following measurements give the dimensions, volume, and weight of burned shale brick from which the fire shrinkage was calculated:

	Dimension in inches	*cu. inches*	*pounds*
Standard shale brick, Cleveland Brick and Clay Co.	4 by $8\frac{7}{8}$ by $2\frac{5}{8}$	92.77	7 4/16
Cleveland Hydraulic Press Brick Co.	4 by $8\frac{1}{2}$ by $2\frac{1}{2}$	85.00	6 6/16
Deckman-Duty Brick Co.	$3\frac{7}{8}$ by $8\frac{3}{4}$ by $2\frac{3}{8}$	81.19	6 14/16
John Kline Brick Co.	4 by $8\frac{7}{8}$ by $2\frac{5}{8}$	92.77	7 7/16
Metropolitan Paving and Brick Co.	$3\frac{7}{8}$ by $8\frac{1}{2}$ by $2\frac{1}{2}$	79.82	7 1/16
Newburg Brick and Clay Co.	$3\frac{7}{8}$ by $8\frac{3}{8}$ by $2\frac{5}{8}$	85.11	7
Average volume		86.11	Average weight 7

Compared with the volume and weight of a dried brick which were 111.28 cubic inches and 7 12/16 pounds, respectively, it was found that the burned brick showed a loss of 25.17 cubic inches or 22.59 percent. The loss in weight was 12 ounces or 9.67 percent.

4 inch paving block,		
Cleveland Brick and Clay Co. 4⅛ by 8¾ by 3½	126.00	10
Deckman-Duty Brick Co. 3¾ by 8¾ by 3½	114.82	10 1/16
John Kline Brick Co., 4 by 9⅛ by 3⅜	123.17	10 7/16
Metropolitan Paving and Brick Co., 4⅛ by 8⅝ by 3½	124.52	9 14/16
Newburg Brick and Clay Co., 4 by 8¾ by 3½	118.48	10 6/16
Average volume	121.45	Average weight 10.15

The average volume of two dried block was 164.34 cubic inches which showed a fire shrinkage of 32.89 cubic inches or 21.31 percent. The average weight of two dried brick was 11 pounds which gave a loss of 13.6 ounces or 7.72 percent.

5 inch paving block,		
Cleveland Brick and Clay Co. 4⅞ by 8¾ by 3⅜	142.75	12 11/16
Deckman-Duty Brick Co., 4¾ by 9 by 3⅜	154.95	13
John Kline Brick Co., 4⅞ by 9 by 3⅜	148.07	12 12/16
Metropolitan Paving and Brick Co., 4¾ by 8⅝ by 3⅜	138.26	12 3/16
Newburg Brick and Clay Co., 4⅞ by 8⅜ by 3½	142.87	11 13/16
Average volume	145.38	Average weight 12.41

The volume of a dried block was 181.92 cubic inches which showed a loss of 36.54 inches or 20.08 percent. The weight of a dried block was $13^{9}/_{16}$ pounds which gave a loss of 1.15 pounds or 8.48 percent.

Compared with the green bricks as they came from the cutting table or repress machine, it was found that the burned product lost the following volume and weight:

FIG. 5

Standard shale brick

Standard shale brick,			
Volume green	117.56 cubic inches,	weight green,	9 pounds
Volume burned	87.11 cubic inches,	weight burned,	7 pounds
Loss	31.45 or 26.75 percent		2 or 22.22 percent
4 inch paving block,			
Volume green	167.90	weight green	12.75
Volume burned	121.45	weight burned	10.15
Loss	46.45 or 27.66 percent		2.60 or 20.39 percent
5 inch paving block,			
Volume green	197.75	weight green	15.00
Volume burned	145.38	weight burned	12.41
Loss	52.37 or 26.48 percent		2.59 or 17.26 percent

Since vitrified paving material is quite expensive, it is subjected to abrasion and absorption tests. The City of Cleveland and Cuyahoga County require that the abrasion test shall show a loss which shall not exceed 20 percent. Some tests show a loss as low as 14 percent in abrasion, while the absorption in 130 tests ranged from 1.6 to 3.4 percent. It is rare that tests for compressive strength are made but two 4 inch cubes of paving block were tested for the Cuyahoga County Engineer's Office with the following results:

	Cracked at	*Broke at*
4 inch cube No. 1	15,708 pounds per sq. in.	19,144 pounds per square inch
4 inch cube No. 2	17,671 pounds per sq. in.	20,616 pounds per square inch

It requires from 40 to 65 men to run a shale brick plant which produces from 40,000 to 55,000 standard brick daily.

Although the various kinds of hollow ware are made by the stiff-mud process, there are two operations which differ from the method of making shale brick. The clay is tempered in wet pans instead of pug mills, and in molding, a sewer-pipe press is used instead of auger machines. Some of the advantages of hollow ware are that a smaller amount of material is used which decreases the cost of production and transportation. In addition

to these factors, they possess sufficient strength for nearly all purposes and afford ample protection against moisture, heat, and cold. Some experiments on the crushing strength of hollow block have recently been conducted at Case School of Applied Science. One block tested for the Camp Conduit Co., by Mr. H. D. Pallister measured approximately 4 by 4 by 12 inches and possessed two webs about half an inch thick, making three hollow spaces. This was tested on the 4 by 12 surface which was the weakest, since it was supported only by two vertical walls. Complete failure of the block took place only after 734 pounds per square inch of pressure had been applied. Considering the amount of available web section, this would make about 3,416 pounds per square inch. Another block 8 by 12 by 12 inches was tested on an 8 by 12 surface. This block had four holes and was supported by three webs. It failed at 740 pounds per square inch of surface, or 4,215 pounds per square inch of available web section.

Another elaborate series of experiments on crushing and absorption were conducted at Case for the Ohio Clay Co., by Professor R. G. Dukes and Mr. H. D. Pallister. The blocks measured approximately 5 by 8 by 12 inches, and had one web giving two cavities. The exterior walls and webs were about one-half inch thick. Twenty different blocks were tested on the 8 by 12 surface which crushed at loads ranging from 94,000 to 177,000 pounds with an average of 113,602 pounds. The crushing load per square inch of area varied from 940 to 1803 pounds with an average of 1210 pounds. Calculated into pounds per square inch of available web section, the crushing loads ranged from 3,752 to 7,437 pounds with an average of 4,871 pounds. As a result of these tests the Building Code of the City of Cleveland was amended so that hollow blocks might be loaded 200 pounds per square inch of available web section. This allows for the usual factor of safety of about 20 percent. The absorption was also tested on twenty of the blocks mentioned previously and ranged from 2.6 to 10.9 percent with an average of 8.8 percent. Incidentally, this shows that hollow ware is not so hard burned, and, therefore, not so completely vitrified as shale brick in which the absorption does not average 3 percent.

THE UTILIZATION OF OHIO CLAY-BONDED SANDSTONES FOR SILICA BRICK MANUFACTURE

BY H. B. HENDERSON AND WILBUR STOUT

The testing of refractory materials began in southern Ohio in 1826 when La Grange Furnace located in Lawrence County was built of sandstone quarried on the hill adjacent to the site chosen. The worth of the native sandstones for such purposes is well established by the fact that they were used in the construction of some 100 charcoal furnaces in what is known as the Hanging Rock iron region. Jefferson Furnace, which is located near Oak Hill in Jackson County and which stands alone as the only active representative of this pioneer industry, was constructed in 1854 of sandstone obtained from a hill some two miles distant. Below the Ferriferous ore and limestone, which constituted the main supply for ore and flux of the furnaces that succeeded best, is found a deposit of sandstone which is very persistent throughout the entire area. This bed was named Hecla sandstone by Dr. Edward Orton, for one of the early and most noted stacks in which it was used was Hecla Furnace, but, due to its correlation with the Pennsylvania rocks, it has since been named Clarion sandstone. This rock was the favorite hearthstone of the charcoal furnace men, and it should thus be credited with forming the smelting pot for much of the early wealth of the area. The refractory qualities of the Clarion or Hecla sandstone have, therefore, been tested for 90 years in all, and it is still used for the hearthstone of Jefferson Furnace after testing its heat resisting properties for some 60 years.

The Clarion sandstone is found in the interval from the Brookville to the Clarion coal, or rather to the Clarion clay, as this bed in southern Ohio lies directly above the sandstone with no sharp line of demarcation separating the two deposits. The thickness of the stratum varies from 20 to 30 feet, but the stone in the upper half is best suited for refractory purposes. This

rock is composed principally of quartz sand with a small amount of clay which, in the main, forms the bonding component. Besides the clay, pyroxene, micas, feldspars, and others are present but the combined amount of these is relatively small, owing to the wide distribution of the Clarion sandstone, to its heat resisting qualities determined by long use in the charcoal furnaces, and to its chemical and physical composition, the writers chose this as one of the sandstones for the manufacture of clay-bonded silica brick. The sample was taken from the quarry of the

FIG. 1

Jefferson Iron Company, located in Section 15, Jefferson Township, Jackson County. The hearthstone ledge, used for many years in relining the furnace, lies near the top of the Clarion member which here is some 30 feet in thickness. The exposed face of the cliff which was about 10 feet in height and 100 feet in length was sampled at regular intervals both vertically and horizontally. The sample so taken constitutes the material used in the tests described in this paper under the designation Clarion sandstone. See Figure 1.

Sandstone with clay for a bonding component are found associated with the Sciotoville clay in Scioto County, Ohio. These beds are somewhat local in distribution and variable in structure and position, but in some localities they have ample thickness for ease in mining. The area in which they are best developed is that near Gephart Station, Scioto Furnace, and Pinkerman in Bloom Township, but they have good volume also at other places. The normal position of these sandstones is at the base of the Sciotoville member, but often layers are found interbedded with the semi-flint clay. They are seldom directly associated with the flint clay unless the semi-flint is absent. The deposit may be represented by a single bed or by this with one or more layers above which are separated by clay. The sandstone below the clay belongs, in fact, to the Sharon conglomerate, the upper part of which in southern Ohio often occurs as a sandstone with but few if any pebbles. Where the Sciotoville clay lies on or close to the sandstones they contain a small amount of clay which constitutes a part of the bonding material. The sandstones interbedded with the semi-flint clay are not a part of the Sharon member, but their composition suggests a common origin at least. These rocks were selected for the second test because they may be mined economically along with the Sciotoville clay, and because their composition is somewhat different from that of the Clarion sandstone used for the first test. The sand is made up of quartz particles admixed with a small number of particles of basic minerals, hence the fluxes in this part of the stone is low. The main bonding component is clay which is in excess of that in the Clarion sandstone, but iron minerals in a finely divided state are also present, although the amount is relatively small. The sample used in the following tests was obtained near Gephart Station, Scioto County, Ohio. Here the sandstone layers interstratified with the semi-flint clay are from 1 to 4 feet in thickness, and they may be mined with the clay. For identification purposes only, it is here called Gephart sandstone.

The interval from the Ferriferous limestone to the Lower Kittanning coal is the most noted clay bearing division of rocks in the geological section of Ohio. This interval contains the

great deposit of Lower Kittanning clay the outcrop of which across southern Ohio from Jefferson County to Lawrence County is distinctly traced by a line of extensive ceramic plants, and the influence of which has been responsible largely for the supremacy of this state in the field of clay products. Along with the clays are also found sandstones whose properties have not been well defined. These sandstones are all cemented with clay the amount of which varies from a few percent to such an amount that the rock ceases to be a sandstone but becomes a siliceous clay. The one chosen and sampled by the writers for the tests given in this paper is high in clay, and it is in marked contrast to the Clarion sandstone used in the first test. These sandstones are found in abundance on the Lower Kittanning clay horizon at almost any point where the member is due. The sample used was obtained from the clay mines in the Oak Hill district, Jackson County, Ohio. The two main components in the mineral are quartz and clay, both highly comminuted. It contains also noticeable amounts of mica and iron oxides. This rock is here designated as Lower Kittanning sandstone.

The Clarion and Gephart sandstones are true ganisters according to the original meaning of the term as used in England, but the Lower Kittanning sandstone is somewhat too argillaceous, although it may be placed in the same class with some limitations.

"The original ganister was a dense, clay-bearing, carbonaceous sandstone of gray, gray-brown or red color, which occurred under the coal measures near Sheffield in England, Dowlais in Wales and in parts of Scotland. The ganister stone from the Lowood mine was found in a bed 10 feet thick at a depth of 1,000 feet, and was considered especially valuable for brickmaking purposes in the early days of the manufacture of silica brick. This ganister was bonded by means of the contained clay impurities and could be formed, dried and burned into brick of fair quality without the admixture of extraneous bonding or fluxing agents."[1]

[1] Havard, *Refractories and Furnaces*, page 31.

Analyses of English ganister is as follows:[2]

	Gartcosh (Scotland).	*Sheffield.*
SiO_2	87.06	88.56
Al_2O_3	11.24	10.16
Fe_2O_3	0.69	0.97
CaO	trace	0.04
MgO	trace	.....
K_2O	0.61	.....
Na_2O	0.33	
Total	99.93	99.73

"German ganister, also called German dinas brick, analyzed as follows:[3]

	RHINE DINAS FOR CUPOLAS	PUDDLE FURNACE SPECIAL BRICK	
	percent.	*percent.*	*percent.*
SiO_2	92.80	90.84	92.85
Al_2O_3	5.94	9.16	4.95
Fe_2O_3			0.87
Fluxes			1.83
	100.00	100.00	100.00

German natural ganister, blocks hewn from Crummerdorf quartz shale, analyzed thus:"

	percent.
SiO_2	88.14
Al_2O_3	6.11
Fe_2O_3	0.45
Alkalies	5.30
	100.00

[2] *Ibid.* page 33.
[3] Havard, *Refractories and Furnaces,* page 50.

The analyses of the Ohio ganisters or clay-bonded sandstones are given in Table I.

TABLE I. ANALYSES OF OHIO SANDSTONES

	CLARION	GEPHART	LOWER KITTANNING
	percent.	*percent.*	*percent.*
SiO_2	88.60	85.39	77.08
Al_2O_3	6.79	9.48	14.62
Fe_2O_3	0.66	1.05	1.00
CaO	None	None	0.27
MgO	0.11	0.06	Trace
TiO_2	Trace	Trace	1.20
Na_2O	0.16	0.52	1.50
K_2O	1.70		
Ignition loss	1.70	3.53	4.26
Total	99.72	100.03	99.93

The above analyses of Ohio clay-bonded sandstones show features of interest when they are compared with those of the foreign materials. The amounts of the various components in the Gephart standstone approach closely those of the respective components in the English ganisters. The total bases determined in the Gephart and in the Scotch Gartcosh sandstones are exactly the same, 1.63 percent. There is some difference, however, between the Clarion sandstone and the English ganisters, for the former contains less alumina and more alkalies, otherwise they are much the same. The composition of the Clarion sandstone approaches that of some of the German ganisters, especially that of the Crummerdorf quartz shale. In this shale the contents, silica 88.14, alumina 6.11, ferric oxide .45 are strikingly close to the contents, silica 88.60, alumina 6.79, ferric oxide .66 in the Clarion sandstone. The alkalies in the shale, however, amount to 5.30 percent, while in the Clarion sandstone they amount to only 1.86 percent. Although the two rocks are dissimilar in the quantities of the respective mineral components present as will be shown later, yet if refractory merits are judged by chemical composition alone, the Ohio sandstone is more refractory than the Crummerdorf shale. In the German ganister used for puddle furnace special brick, the silica is few percent higher, and the

alumina from one to two percent lower than they are in the Clarion sandstone, while the alkalies and ferric oxides are practically the same in both rocks, in fact, the two materials should have about the same heat resisting qualities.

The Lower Kittanning sandstone, taken for the high alumina end of the series, contains some 10 percent less silica, 4 percent more alumina, and 2.50 percent more bases than the original ganister used abroad. In fact, it is on the border between a ganister and a siliceous clay.

No rational analyses were made of the three sandstones used, but some idea of the quantities of clay, feldspathic material, and quartz they contain may be obtained by calculating the alkalies to feldspar, the excess alumina to clay, and the residual silica to quartz. Using this method the results obtained for the three Ohio samples and the foreign sandstones are given in Table II.

TABLE II. MINERAL COMPOSITION OF SANDSTONES

	GARTCOSH (SCOTLAND)	GERMAN PUDDLE FURNACE BRICK	CRUMMERDORF	CLARION	GEPHART	LOWER KITTANNING
Feldspathic material.	6.40	10.83	31.35	11.40	3.07	8.87
Clay base	21.85	6.44	.78	11.87	22.56	32.85
Quartz	70.99	82.36	67.42	75.64	72.91	56.06
Other components...	0.69	.87	.45	0.81	1.49	2.15
Total	99.93	100.50	100.00	99.72	100.03	99.93

When the Clarion sandstone is compared by this method to the Scotch ganister it shows nearly twice as much feldspathic material but only about as much clay base as the foreign stone, while the amount of quartz in the former is not far different from that in the latter. As the feldspathic material is the main fluxing factor in both stones, the foreign ganister is evidently more refractory than the Ohio rock, but the effect of the additional fluxes in the Clarion sandstone is partially offset by its more siliceous nature. It also ranks close to the rock used in Germany for puddle-furnace brick. The feldspathic materials

and iron oxides are about the same in both rocks, but the siliceous matter is higher in the German stone. Although the components shown by analysis in the Crummerdorf and in the Clarion stones are much the same, the mineral components determined by calculation are far different. The Crummerdorf shale is made up almost wholly of comminuted feldspar and quartz with but little clay base.

The quantities of clay base and of quartz in the Gephart sandstone are thus shown to be nearly identical with that in the Scotch ganister, but the quantity of the feldspathic fluxes in the Gephart stone is only one-half that in the foreign material. When the fluxing components only are considered, the Gephart sandstone is equally or even more refractory than either the Scotch or Clarion ganisters. The Lower Kittanning sandstone can scarcely be considered in the same class as the Scotch ganister as the clay base in the former is about 11 percent higher and the quartz nearly 15 percent lower than they are in the latter, although the difference between the quantities of feldspathic matter in the two stones is not large. In composition it is also far different from the other Ohio stones tested.

No information with regard to the texture of the English ganisters is at hand. Since these ganisters are described as clay-bonded sandstones taken from beds associated with coals their texture is probably much the same as that in the Ohio sandstones which are also from deposits similarly placed. When the texture of the Ohio sandstones is judged by the eye, the Clarion is the coarsest grained, and the Lower Kittanning the finest, while the Gephart is nearer the former than the latter. This grading is verified by a screen test which is given below:

MESH	CLARION	GEPHART	LOWER KITTANNING
	percent	*percent*	*percent*
20-40	25.7	5.6	2.7
40-100	58.0	60.7	29.4
100-260	8.5	15.8	26.5
260-	7.8	17.9	41.4

The screen test shows the degree to which the materials have been comminuted. Of the three sandstones, the Clarion is much the coarsest grained and the Lower Kittanning the finest. When the quantity of material in each with sizes between 20-100 mesh are considered, the Clarion, Gephart and Lower Kittanning sandstones are in the ratio of 84:66:32. The quantity of this sized material in the Clarion, and in the Gephart sandstone is approximately the same as that of the quartz and feldspathic material determined by calculation. In the Lower Kittanning sandstone this is not true, for here the quantity of the 20-100 mesh particles only equals about one-half the sum of these body components. Next consider the finer parts or that below 100 mesh size. The clay bases in the Clarion and Gephart sandstones include the material which went through the 260 mesh screen, and one-half that between 100-260 mesh size. In the Lower Kittanning sandstone the clay base equals only about three-fourths of the material that passed the 260 mesh screen, and only about one-half of that finer than 100 mesh. As the physical state of a mineral aggregate is an important factor in the fusion behavior of the body, the main points brought out by the screen test are listed below and considered later under heat treatment.

(a.) The quartz and feldspathic material in the Clarion and Gephart sandstones are rather coarse grained and well sorted. The quantity of fine material is low and about equals that of the clay base.

(b.) The quartz and feldspathic material in the Lower Kittanning sandstone is finely divided and not differently separated. The clay base is admixed with about an equal quantity of this finely divided granitic material.

(c.) Physically the Clarion and Gephart sandstones are closely related, but these are widely separated from the Lower Kittanning.

The clay-bonded sandstones were made into ware by the most common method used in brick making, that is, by simply crushing the materials which were then shaped into ware by hand molding. The samples were then treated as follows:

Clarion sandstone: The sample of ganister was put through a jaw crusher which reduced it so that the largest pieces were

about 3/8 inches in diameter. This crushed product was then made thoroughly wet and allowed to stand over night so that it would come to an even temper. It was next formed into ware in a standard fire-brick mold. No particular difficulty was experienced during the process, but the material was very tender; hence it required careful treatment. The material was also tried in the dry press, but it did not work well by this process, for the brick were too fragile to withstand handling without crumbling. Owing to the porous nature of the brick made by either method, they may be dried rapidly and safely in almost any type of dryer. The hand molded brick after drying, although somewhat tender, withstood handling, and they had sufficient strength to support the kiln load if not set too high. While the working qualities of the Clarion sandstone are not all that may be desired, with only a small addition of clay at most, it may be manufactured on a commercial basis into marketable ware.

Gephart sandstone: The sample of Gephart sandstone was treated in the same manner as the former. The tempered material was easily shaped into ware either by hand molding, or by the dry press process, as it had considerable plasticity or bonding power. The dried ware had sufficient strength to withstand the handling demanded in brick manufacture, and to withstand the load attendant with setting. The working qualities of the Gephart sandstone are such that it may be made into a good clay-bonded silica brick without the addition of extraneous material.

Lower Kittanning sandstone: The Lower Kittanning sandstone was made into ware by the same method used with the preceding samples. The tempered material had sufficient plasticity to be molded in much the same manner as soft-mud fire brick are formed. The bonding power of the clay in the mass was almost enough to allow the material to be made into ware in a stiff-mud brick machine. This body worked very good in the dry press also. The strength of the dried ware was about equal to that of an ordinary fire brick in the same condition, hence the brick are well able to withstand the handling and load strains to which they are subjected while in the green state. The test shows that the Lower Kittanning sandstone may be manufac-

tured into ware with good structure and shape by most any of the common methods used in making refractory brick.

Burning Behavior. Brick made from each sandstone were burned to cone 12 or approximately 2500° Fahr. in a gas fired kiln. Measurements of the brick in the dried and burned states are given below:

	LENGTH OF DRIED BRICK	LENGTH OF BURNED BRICK	DIFFERENCE IN PERCENT
Clarion	9 inches	$9\frac{1}{4}$ inches	2.8 expansion
Gephart	9 inches	$9\frac{3}{8}$ inches	1.4 expansion
Lower Kittanning	9 inches	$8\frac{3}{4}$ inches	2.8 shrinkage

The difference in linear changes shown in the above test of the three samples corresponds in a general way to the amounts of clay contained. The Clarion brick acts like a true lime-bonded silica brick, while the Lower Kittanning sample has a shrinkage factor much the same as that of ordinary clay ware.

The brick from the Clarion sandstone at cone 12 has a body structure not unlike that of the original sandstone, a little more friable and porous perhaps but resembling it closely in color and physical appearance. The Gephart sandstone produced a brick much harder than the original sandstone and more firmly cemented than the brick from the Clarion rock. It has a hardness close to that of lime-bonded silica ware. The color of the sandstone is dark gray while that of the brick is light buff. This change in color is due to the oxidation of the finely divided iron from the ferrous state in the stone to the ferric state in the brick. Vitrification of the clay bond has progressed to such an extent that the sand grains are held quite firmly, but no body vitrification is apparent, for the brick still has a marked granular structure when examined under the microscope. The ware from the Lower Kittanning sandstone vitrified at this temperature to a firm dense body with a stony fracture. It is very dissimilar in physical appearance from the original material. This ware is capable of withstanding severe abrasion as well as high temperatures.

A piece of the Clarion sandstone taken from the hearth of old Jefferson Furnace shows the effects of high temperatures, and of slag action. On the outer surface or the face exposed to the furnace action there is a very thin layer of slag which has not penetrated the stone, but is merely a surface coating. Next there is a vitreous, slightly dark layer, about ⅜ of an inch in thickness in which the sandstone has undergone a complete transformation, while the remainder of the rock remains unchanged.

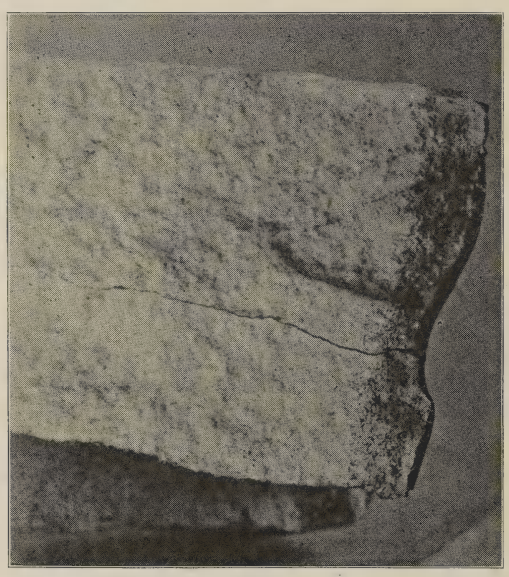


FIG. 2

A microscopic examination of the vitreous layer was made by Dr. McCaughey who reports as follows:

"The sample as examined was prepared by removing all visible traces of slag and the white (unaltered) rock. A portion of the gray rock was then prepared so that it would pass a 100 mesh sieve. A slide was made of another portion.

"The gray stone (product of alteration) was found to consist largely of tridymite (SiO_2) and about ten percent original quartz grains. It is presumed that the quartz came from the

portion of the rock closest to the unaltered rock, since no trace of quartz was found in the thin section.

"The index of the grains were found to be between 1.47 and 1.48, also these showed aggregate polarization with pale birefringence colors and negative elongation ($-c$) which is quite characteristic of tridymite.

"The rock section shows that there has been little slag penetration. Index of refraction of slag=1.50. The tridymite aggregates retain the original form of the quartz grains constituting the sandstones. (See Fig. 4). In fact this structure

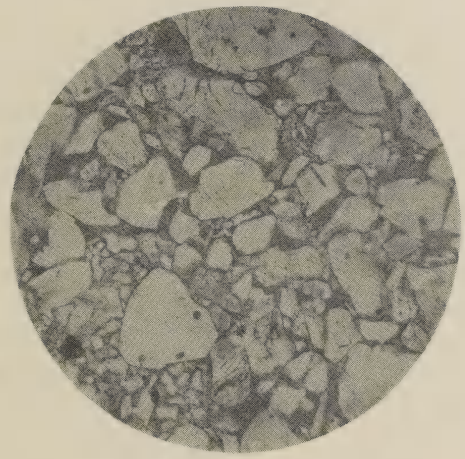


FIG. 3. Clarion Sandstone.

might be called pseudomorphic. The change in mineral composition is most striking, considering the fact that there is little change in the granular character of the rock.

"Quartz has a specific gravity of 2.65, tridymite 2.3, so that one may see considerable expansion has taken place. The change in volume is evidenced by the shattering of the original grain, constituting the rock.

"I might note that cristobalite is the stable form of SiO_2 above 1470°, between 1470° and 870° tridymite, below 870 quartz. The index of cristobalite is 1.484 and 1.487. In no case

did I find grains index above 1.48, except the original quartz grain."

"Figure 3 shows a micro-photograph of a thin section of the original rock (Clarion sandstone). Figure 4 is the micro-photograph of the rock as altered by use in the iron furnace. A comparison of these two photographs will show that the original rounded character of the grains is preserved in Figure 4 and also the shattering due probably to expansion."

Refactory Test. Small cones made from each of the three samples were subjected to a heat test using standard Seger cones

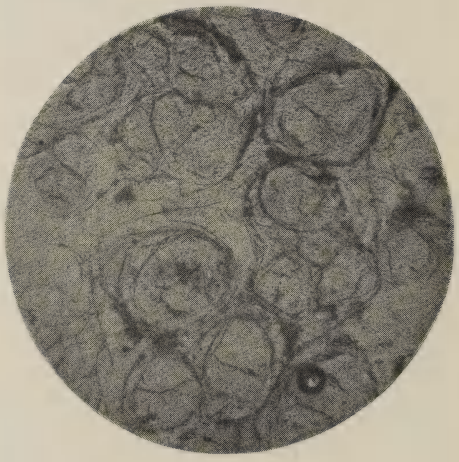


FIG. 4. Furnace Hearth Stone.

for the indicator of refractoriness. The tests were made in a gas-fired furnace, and the results obtained are given below:

	FUSION POINT		
	CONE	° FAHR.	° CENT.
Clarion	30	3,148	1,730
Gephart	30	3,146	1,730
Lower Kittanning	29	3,110	1,710

These tests show that the three clay-bonded sandstones from the coal formations of Ohio are all within the limits demanded for refractory material that small variations in the quantities of flux and clay base in highly siliceous materials have but little effect on the final fusion point, although they may change greatly the physical state of the body, and that the fusion points of these sandstones are close to that of pure quartz.

Uses of Ohio Clay-Bonded Sandstones. The Ohio ganisters have a possible wide range of usefulness, although so far they have had but little application in metallurgical or other high temperature operations, except when used for charcoal furnace linings. Ware made from these sandstones is well fitted for linings of puddle furnaces, cupolas, ladles, annealing ovens, and Bessemer converters, for building both beehive and retort coke ovens, coal-gas retorts, glass furnace walls, lime and ceramic kilns, and for boiler settings, regenerator checkers, and crucibles for acid slags.

General Conclusions. There are present in the coal formations of Ohio, clay-bonded sandstones which are well within the limits of refractory materials. In general properties these sandstones or ganisters compare favorably with the original English ganisters, or with those used extensively in Germany. These Ohio ganisters may be made into commercial ware without the addition of extraneous material, and by the common methods now in use in ceramic practice.

SOFTENING POINTS OF POTASH FELDSPAR-STEATITE MIXTURES[1]

BY W. S. HOWAT, PITTSBURGH, PA.

In attempting to develop a number of porcelain bodies, it was thought desirable to work up several in which steatite had been introduced. It was intended to obtain some of the advantages of steatite such as its influence in promoting translucency, its tendency to whiten the body, and its promotion of slow vitrification, as suggested by Parmelee[2].

It was also hoped to improve the heat resisting qualities of the bodies.

It was in endeavoring to answer the question as to just how much steatite was required to intensify the fluxing action of the feldspar to a maximum that the present work was undertaken. It was decided to answer this question by determining the softening temperatures of a series of feldspar-steatite mixtures.

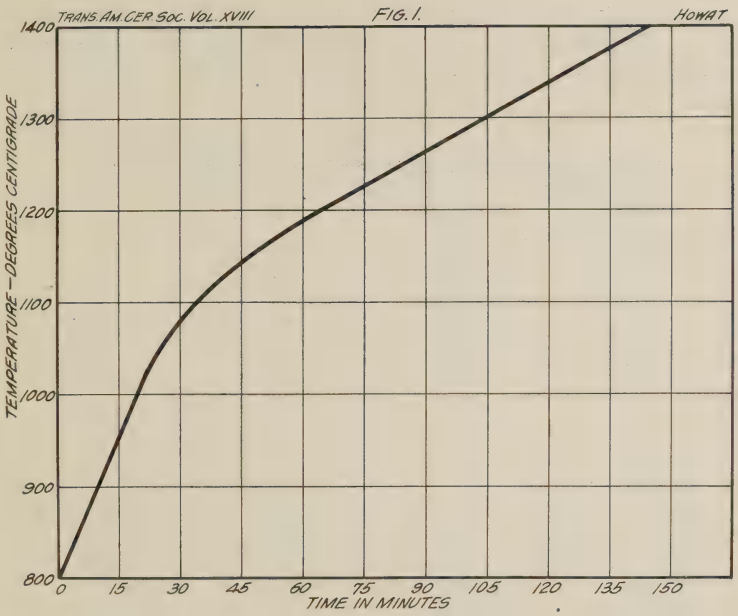
A 100 gram sample of each mixture was weighed out and ground dry for four hours in a small ball mill. Small cones (7/32 inch at base and 1 inch in height) were then molded from each mixture with the aid of detrin as a binder.

For heating the cones a small electric furnace of the platinum resistance type was used, the inside diameter of the furnace being approximately 1½ inches. The cones were inclined at an angle of 60° towards the front of the furnace, the thermocouple used being inserted from the back, with the junction about 1/16 inch from the cone.

Temperatures were obtained by using a Leeds and Northrup potentiometer graduated 0.1 millivolt and accurate to 0.02 millivolts.

The furnace was heated rapidly to 800°C and then regulated to follow the time-temperature curve shown in Figure 1. The rate of heating above 1150° was 2½° per minute.

[1] By permission of the Director, Bureau of Standards.
[2] *Trans. Amer. Cer. Soc.*, Vol. XV.


TRANS. AM. CER. SOC. VOL. XVIII
FIG. 1.
HOWAT
TEMPERATURE - DEGREES CENTIGRADE
1400
1300
1200
1100
1000
900
800
0 15 30 45 60 75 90 105 120 135 150
TIME IN MINUTES

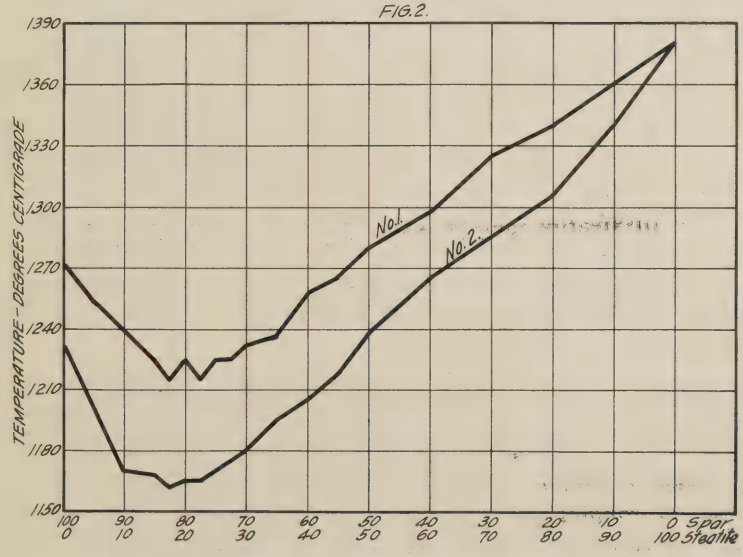


FIG. 2.
TEMPERATURE - DEGREES CENTIGRADE
1390
1360
1330
1300
1270
1240
1210
1180
1150
No. 1.
No. 2.
100 90 80 70 60 50 40 30 20 10 0 Spar.
0 10 20 30 40 50 60 70 80 90 100 Steatite

Two series of mixtures were run, in the first, using Maine feldspar and in the second, a sample obtained by pulverizing a selected sample of microcline. Analyses of these and of the steatite are given in Table I.

TABLE I. CHEMICAL ANALYSES OF MINERALS USED

	MAINE FELDSPAR	STEATITE	MICROCLINE
SiO_2	68.56	45.84	64.90
Al_2O_3	17.36	1.26	19.04
Fe_2O_3	0.30	0.46	0.13
CaO	0.22	8.40	0.33
MgO	0.12	31.79	0.03
Na_2O	3.64	0.10	1.76
K_2O	6.90	0.30	13.88
Ignition	.52	14.98	0.26

Table II and the accompanying curves (Fig. 2) show the softening points obtained. The temperature taken was that at which the tip of the cone had bent over to touch the pat on which it was supported. Each point is the average of two or more determinations, the maximum variation being not over 5°C. The temperature interval between the initial bending and the final point taken was approximately 15°C.

The values of the softening points as here given are, of course, arbitrary, such a series of mixtures having no true melting points, being dependent upon various factors, such as size of cones used, rate of heating, fineness of materials, etc. For example, there was a difference of 28° between the softening point of the Maine feldspar and a sample of the same material obtained by stirring in water and removing the finer material held longest in suspension.

The mixtures of feldspar and steatite having the lowest softening point, however, was found regardless of the arbitrary nature of the softening points themselves, and it was this mixture we wished to obtain. With both samples of feldspar, percentages of steatite ranging from 17½ to 22½ percent have about equal effects in lowering the softening temperatures to a minimum. In the case of the commercial feldspar the minimum temperature

was 1215°C and for the selected sample 1160°C, as compared with the softening temperatures of the feldspars alone of 1272°C and 1232°C respectively. The eutectic composition given above should, therefore, prove most useful in using a mixture of these materials in vitreous bodies.

TABLE II. DEFORMATION TEMPERATURES

FELDSPAR	STEATITE	SOFTENING TEMPERATURE, MAINE SPAR	SOFTENING TEMPERATURE, MICROCLINE
percent	*percent*	*degree C.*	*degree C.*
100	0	1272	1232
95	5	1250	1200
90.	10	1238	1170
85	15	1225	1168
82½	17½	1215	1160
80	20	1225	1165
77½	22½	1215	1165
75	25	1225	1170
72½	27½	1225	1180
70	30	1232	1182
65	35	1237	1195
60	40	1258	1205
55	45	1265	1218
50	50	1280	1238
40	60	1297	1265
30	70	1325	1285
20	80	1340	1305
10	90	1360	1340
0	100	1380	1380

SLAG PAVING BRICK

BY E. T. MONTGOMERY AND P. W. BURDICK, ALFRED, N. Y.

We can, today, make very good clay pavers, but at our best we have not yet been able to make a clay block which will stand the wear and tear under heavy city traffic as well as a granite block. The opinion has been expressed, however, by some paving brick manufacturers that this is due to the greater depth or thickness of the granite pavement.

In making slag paving brick from an igneous fusion of suitable clays and limestone or dolomite, with perhaps small quantities of additional flux, we are but attempting to produce a synthetic igneous rock.

By-product slag block[1] have for a long time been made in small quantities, and when carefully made and annealed have given good account of themselves as a paving material, both in this country and abroad. The best of these slag brick have been made from copper smelter slag, having the following approximate composition: SiO_2 47-57 percent, Al_2O_3 5-18 percent, Fe_2O_3 3-10 percent, CaO 18-33 percent, and MgO 1-8 percent. The greatest drawback to the by-product production of slag brick is, of course, the limited opportunity for control of the slag composition.

It was for the purpose of developing data upon which an independent industry might be based, that the present investigation was undertaken. The work to be here reported is a continuation of that done at the Pittsburgh laboratory of the Bureau of Standards as reported on page 547, Vol. XV of these *Transactions*. The conclusions reached in the Bureau of Standards work were as follows: "A suitable composition for slag brick should not contain less lime and magnesia than that represented by a slag composed of 52 percent dolomite and 48 percent shale.

[1] *Cements, Limes and Plaster*, Eckel, p. 685.

That dolomitic slags, as a class, show greater fluidity, crystallize more readily and seem superior in hardness and toughness to the limestone slags."

From this as a starting point it was our object, first, to try to lower the fusion points of the best slags developed in the above cited investigation, since they are all rather high for commercial furnace operation, by the addition of such quantities of the cheaper fluxes as seemed commercially feasible. Second, to compare briquettes of these various compositions with a standard clay paving brick, by rattler test; and, third, to finally make full size bricks from the best composition and test them by the standard rattler test against a well known clay paver.

The composition of the raw materials used are given in the following table together with those used at the Bureau of Standards, for the purpose of comparison.

TABLE I. CHEMICAL COMPOSITION

COMPONENT	DOLOMITE	BU. OF STD. DOLOMITE	SHALE	BU. OF STD. SHALE
	percent	*percent*	*percent*	*percent*
Silica	.94	.46	53.20	59.72
Alumina	.15	.23	23.25	19.28
Ferric oxide	.45	.13	10.90	7.99
Manganese dioxide	.....	.....	.52	.....
Titanium oxide	.....	.....	.91	.....
Lime	.....	.....	1.01	1.21
Magnesia	.....	.....	.62	1.77
Calcium carbonate	54.09	54.10	.....	.....
Magnesium carbonate	44.58	45.44	.....	.....
Soda	.....	.....	2.69	.47
Potash	.....	.....		3.06
Loss on ignition	.....	.....	6.80	6.96
	100.21	100.28	99.90	100.46

Two different dolomitic limestones were furnished us through the courtesy of the Charles Warner Co. of Cedar Hollow, Pa. and the Woodville White Lime Co., of Woodville, Ohio. Both were of nearly the same composition and the Charles Warner Co. stone was the one used in the work. The shale used was the local Devonian shale which outcrops at Alfred.

As the composition lowest in dolomite which gave a satisfactory slag in the Bureau of Standards work, had been placed at 52 percent dolomite and 48 percent shale, the following series was made up into small cones and their deformation points determined in an electric furnace.

TABLE II. DEFORMATION TEMPERATURES IN DEGREES C.

	No. 1	No. 2	No. 3	No. 4	No. 5	No. 6	No. 7	No. 8
Dolomite	52	56	60	64	68	72	76	80
Shale	48	44	40	36	32	28	24	20
Temperature of deformation	1220°	1200°	1240°	1280°	Above 1300° C.			

As mixture No. 2, though deforming at 1200° C., had in the Bureau of Standards work required a temperature of 1320° C. to cause fusion of a batch in a large crucible, it was thought best to confine our attention to mixtures No. 1 to No. 4, those higher in the series being too infusible to be commercially available. Having now determined the deformation temperatures for these four mixtures, various percentages of several different fluxes were added to each, the batches ground and put through an 80 mesh sieve, and cones again made up from each batch for the determination of deformation points. Table III shows the fluxes used and the deformation temperature for each mixture.

From the table it will be seen that the most effective flux was 10 percent of the fluorspar-cryolite eutectic added to mixture No. 3 consisting of 60 parts dolomite and 40 parts of shale. This mixture No. 3 was therefore selected as the basis for all future tests. Large batches of No. 3 with additions of 10 percent and 20 percent of the fluorspar-cryolite eutectic, 10 percent sodium carbonate and 20 percent Albany slip were now made up, melted in large crucibles and poured into two-part cast iron molds, forming a briquette 4 in. by 2 in. by 1½ in. To avoid cracking, the molds were heated up to between 800 and 900° C. in a suitable furnace before the slag was poured into them. After casting, the temperature was held at 900° for about two

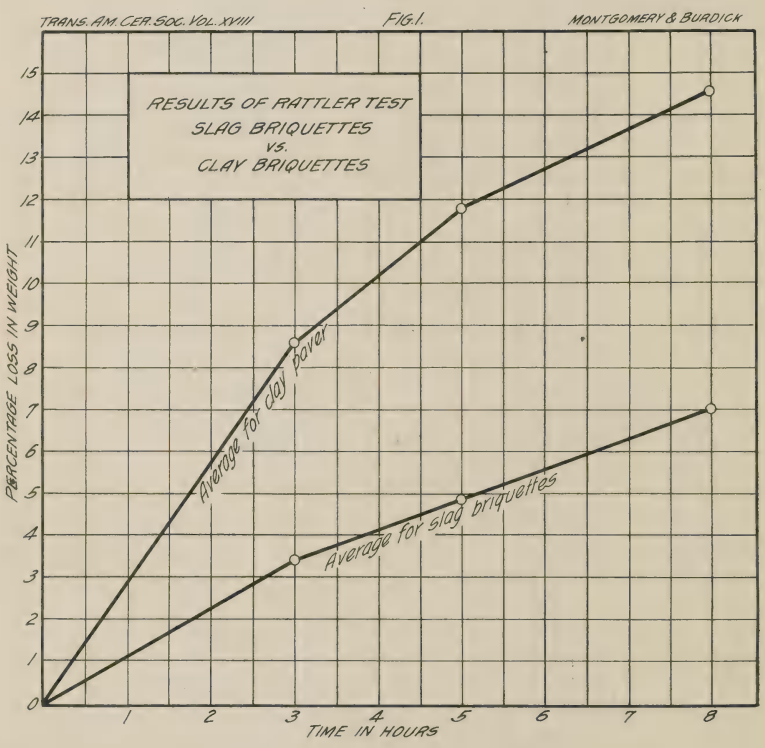
TABLE III. DEFORMATION TEMPERATURES IN DEGREES C.

FLUX ADDED TO MIXTURES	1120°	1140°	1160°	1180°	1200°	1220°	1240°	1260°	1280°C
	MIXTURE NUMBER								
Dolomite shale mixtures....	—	—	—	—	No. 2	No. 1	No. 3	—	No. 4
WITH									
5 percent Fluorspar-cryolite-eutectic[1]	—	No. 3	No. 2	No. 1	—	No. 4	—	—	—
10 percent Fluorspar-cryolite-eutectic[1]	No. 3	—	No. 2	No. 1	No. 4	—	—	—	—
10 percent Na_2CO_3.....	—	—	No. 2	—	No. 1	No. 3	—	No. 4	—
10 percent Soda spar.....	—	—	—	No. 3	No. 2-1	No. 4	—	—	—
10 percent Albany slip.....	—	—	—	—	No. 2-1	No. 3	—	—	No. 4
20 percent Albany slip.....	—	—	—	No. 2	No. 1	No. 3	—	No. 4	—

[1] NOTE — Eutectic above consists of 65 percent fluorspar (CaF_2) + 35 percent cryolite (Na_3AlF_6) and melts at 910° C.

hours, and then the furnace was allowed to cool slowly. In this way good, strong, tough, annealed and stony briquettes were always obtained. Before using, the molds were washed on the inside with a clay-alumina mixture which caused the briquettes to release from the molds easily.

The briquettes were then rattled for one hour against pieces



of Metropolitan paving block, broken to the same size as the briquettes, in a ball mill with a suitable charge of pebbles. All of the samples had the sharp edges and corners ground off on an emery wheel before going into the rattler. The following table shows the results averaged from three samples of each mixture.

20 percent fluorspar-cryolite briquettes. 4.88 percent loss
10 percent fluorspar-cryolite briquettes. 5.14 percent loss
20 percent Albany slip..... 5.46 percent loss
10 percent sodium carbonate..... 6.02 percent loss
Paving brick samples ..... 7.85 percent loss

It should be noted here that the lowest melting mixtures also give the best rattler test.

Since the difference in the rattler test between the slag with the 20 percent fluorspar-cryolite flux and the one with 10 percent, was not enough to justify the use of the larger amount on account of increased cost, more briquettes were made of dolomite-shale mixture No. 3 plus 10 percent fluorspar-cryolite flux. It was found that with this composition fusion could be obtained at 1250° C, if time enough were allowed, but that the batch could be melted much more easily at higher temperatures.

A number of these briquettes were rattled against briquettes of a similar size and weight, obtained from a Dunn-wire-cut-lug Metropolitan shale paver, taken as a standard for clay pavers. These were rattled for eight hours, weighed and the loss calculated at the end of three, five and eight hours. The result is shown on curve sheet, Figure 1. The curves represent averages of three samples in each case.

These results seem to point to the superiority of the slag paver in the rattler test. It has also been shown that the temperature of fusion can be materially reduced by the addition of small amounts of suitable fluxes. Dolomite-shale mixture No. 3 is probably superior to either No. 1 or No. 2 because somewhat higher in dolomite, hence giving a more stony slag. Ten percent of the fluorspar-cryolite flux reduced the deformation temperature of this mixture from 1240° C. to about 1120° to 1130° C. and caused the fusion of a large batch at 1250° C. which is approximately the deformation temperature of this mixture without the flux.

Attempts to make full size slag paving bricks proved unsuccessful in the time available. Crucible methods of melting the

slag proved inadequate for the large quantity required, and the lack of time prevented the construction of a tank furnace.

No cold junctions were used for the temperatures given in this paper, and no corrections therefore have been made. Add 7° or 8° C. for corrected readings.

A MEANS FOR THE DETERMINATION OF THE FINENESS OF RAW MATERIALS

BY F. W. WALKER, JR.

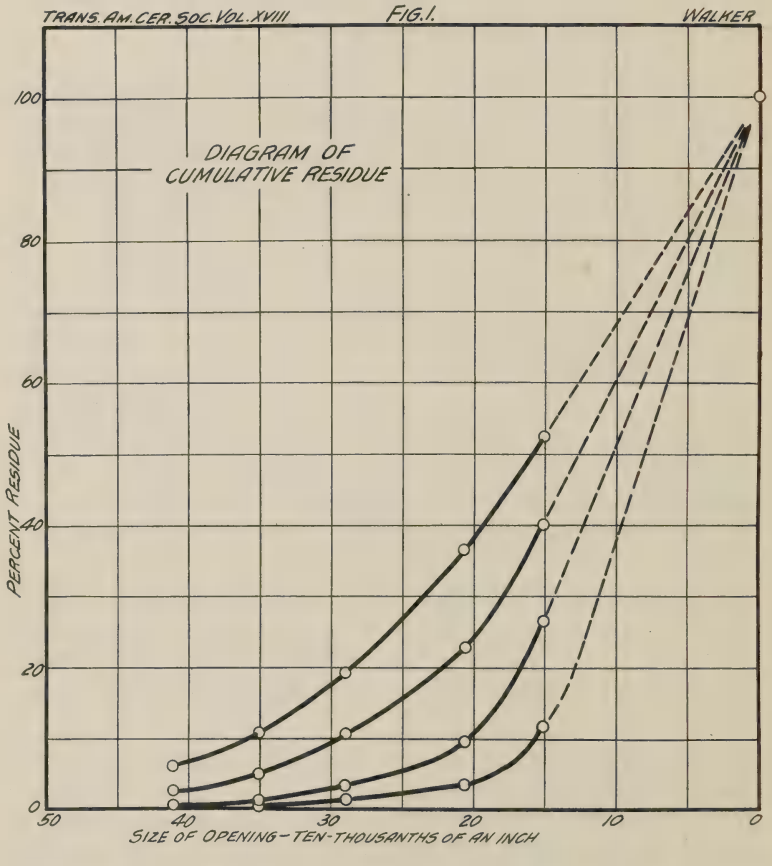
The lack of a standard method for the comparison of the degree of subdivision of raw materials is a big handicap to the potter, as well as to the grinder of flint and feldspar. Many of the difficulties encountered in potting can be attributed to variation in raw material, and ceramic progress will be more or less limited until this fundamental is given more careful study.

The many difficulties in the way of bringing about a standard for testing this physical property have been the principal cause of present conditions, but considerable progress is being made, especially in connection with the use of sieves, and the establishment of a standard method for comparison by their use, seems quite possible. The value of any test for this work should be based largely upon its ability to correctly classify the various sizes of grain and, especially, that which constitutes the larger proportion of the ceramic materials, namely, .0015 inches and less in size.

The elutriation processes which have been used quite extensively in making measurements of this kind, are in some ways well suited to this work, but the difficulties encountered in their operation are so numerous as to practically eliminate them from use by the practical ceramist.

The only test at the present time, that seems to be acceptable to the potter is the determination of the amount of residue on some one sieve, usually the 140 mesh. This test is referred to quite frequently but very seldom in connection with sufficient data to judge the size of opening or the sieving process. The value of such a test is almost useless and, under the best of conditions, could only prove of limited value, because the small amount of residue contained thereon, would give little information regarding the character of the bulk of the material.

The advisability of using a method of this kind, if standardized, is quite evident when considered in connection with the cumulative direct plot as referred to by Mr. Kerr in Vol. XV. The general character of the cumulative curve for any given pro-

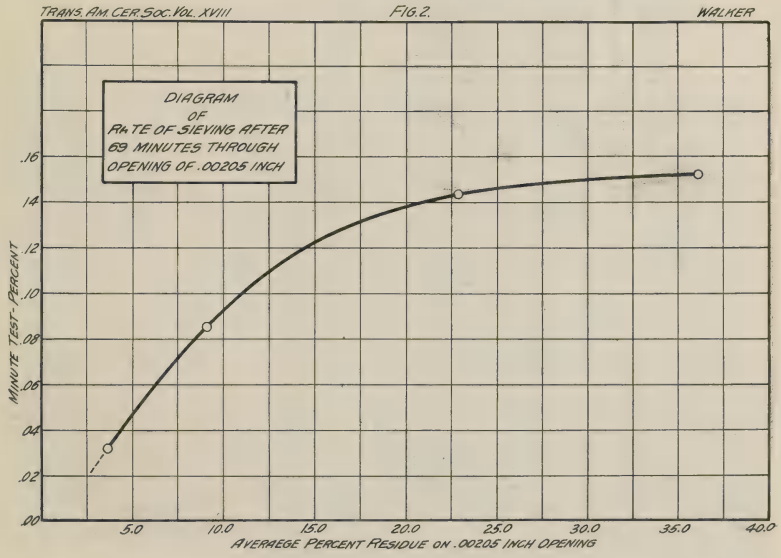


cess of grinding, is fairly constant, and the determination of some one point on the curve is sufficient to give a fair idea of its location in the field.

In as much as the Bureau of Standards, at Washington, has recently recommended a standard 200 mesh sieve, as well as sieving process for the testing of cement, an investigation was car-

ried out in accordance with their specification, hoping to be able to fix a point on the cumulative curve with a fairly high degree of accuracy and, furthermore, should such prove satisfactory, to bring the same test into common use for the potters.

The results of the investigation were only partially satisfactory for it was found that the amount of residue was small and that a variation of .5 percent from the mean value could be expected for residues up to 15 percent. This difficulty is belived attributable to the large amount of flour, and it is quite possible



that a modification of the government process could be worked out to give better results, but the recent development in the manufacture of fine mesh sieves of fairly constant proportions has made possible more detailed comparison.

As the result of this development, further researches were undertaken using a series of sieves inclusive of the 280 mesh based on an opening of .0029 inch and varying in the ratio of the fourth root of two or 1.189, the factor recommended by Prof. Richards in his work on ore dressing.

The following test was outlined after some preliminary

study, and its merits determined by a comparison of the results obtained, using a single material ground in a cylindrical mill for different lengths of time, thus securing data for various degrees of fineness which more than covers the field of the ceramist. Ten samples of each grinding were carefully taken by means of a riffler.

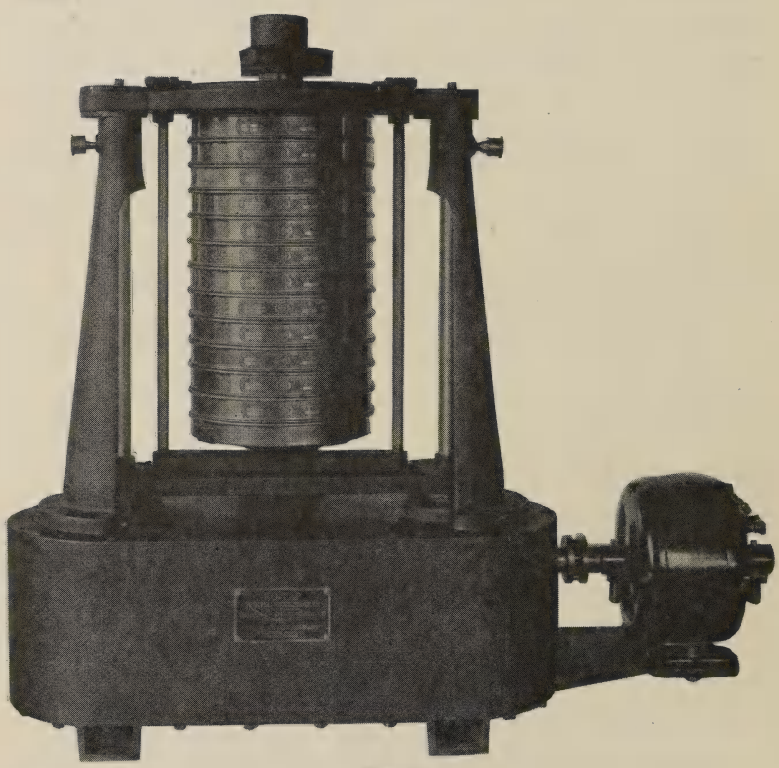


FIG. 3

Place a 100 gram sample of the material to be tested, the same having been dried to 100°C, in a large beaker and after thoroughly mixing with water, decant onto a 280 mesh lawn 8 in. W. S. Tyler Standard Screen Scale and carefully wash by submerging just so that the water will cover the product in the sieves, the wave like motion of the water washing the material through the sieves as it moves back and forth. This must be continued

until all the fines have been removed as evidenced by the lack of suspended material when washed with clear water. Following this operation, the sieve is allowed to drain, placed over a ten inch watch glass and put in an electric oven, heated to 75°C and allowed to remain until thoroughly dry.

Much of the residue which when wet is retained on the 280 mesh sieve, passes through readily when dry and therefore care must be used to avoid losses.

The sieves to be used, namely, the W. S. Tyler Standard Screen Scale sieves of 115, 150, 170, 200 and 280 mesh with open-

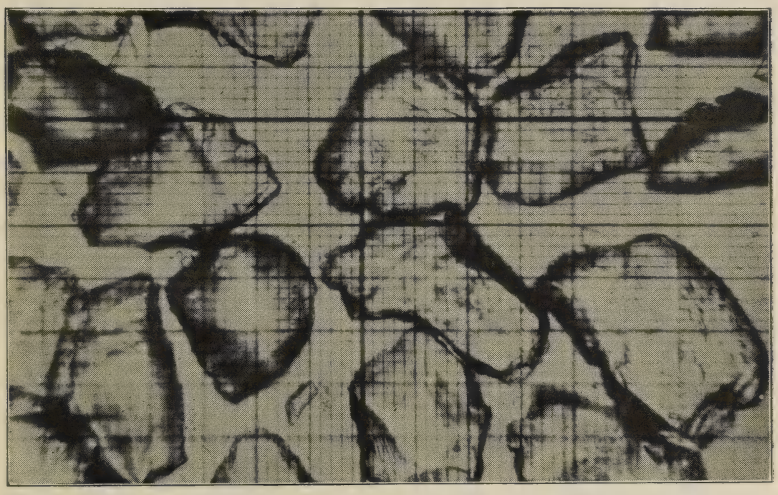


FIG. 4

ings of .0049, .0041, .0035, .0029, and .0020 inches, respectively, are arranged one on top of the other in the order named, the 280 mesh sieve being on the bottom, and the residue brushed from its container onto the 115 mesh sieve. The stack is next placed in the W. S. Tyler Co.'s Ro Tap machine (see Fig. 3), and allowed to remain for 69 minutes, after which a one minute test is made, and the amount of material passing in that time determined. The residue on each sieve, as well as the pan, is removed carefully, using a 1 in. soft brush, weighed, and the residue on all sieves calculated.

The results of the study are shown in Table I, and graphs of the average results are shown in Figures 1 and 2. From these curves a very fair idea of the degree of subdivision can be had, and especially by obtaining an average figure of three or more determinations for that passing the 280 mesh lawn which is found to have an average comparative diameter of .0015 inches. The actual diameter is about 25 percent higher because of the shape of the opening. Figures 4, 5, 6 and 7 are micro-photographs of residues retained on 170 mesh sieve, 200 mesh, 280

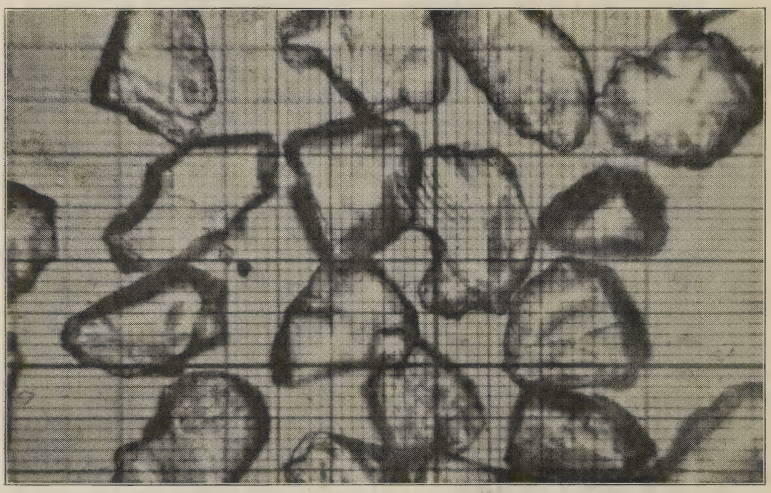


FIG. 5

mesh and through 280 mesh sieve, the fines. Each co-ordinate division has a length of .00833 m. m.

In the preliminary study efforts were made to dispense with the use of a machine but the work proved too laborious.

In concluding it can be said that results of a fairly high degree of accuracy can be had by this method, and this, together with the ease of operation, the elemination of the personal factor, and the better analysis of the results made possible by the different residues, makes the test appear as a possible starting point for the establishment of a standard.

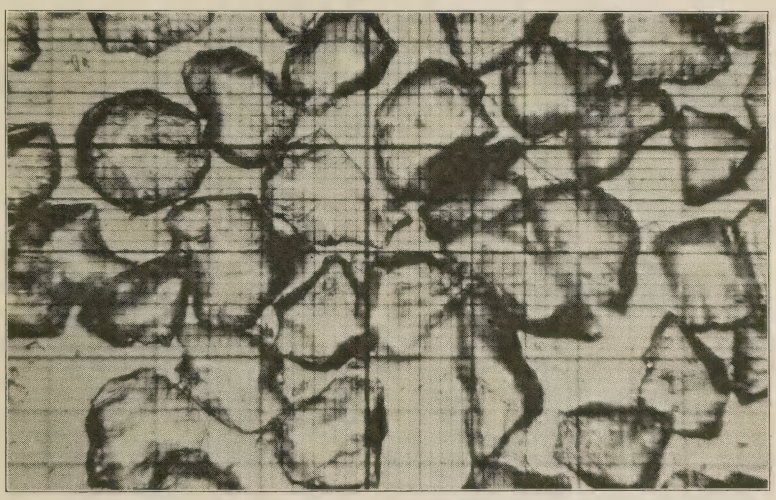


FIG. 6

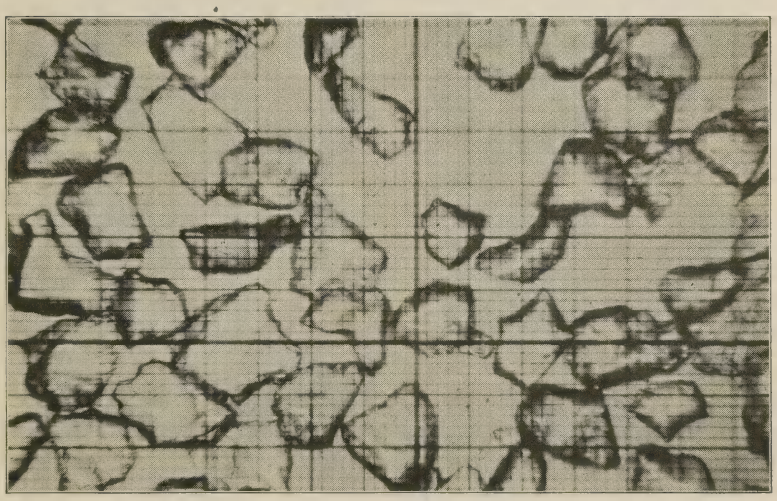


FIG. 7

TABLE I. TEST NO. 1. WHITE SAND. 14 HOURS GRIND

MESH	No. 1	No. 2	No. 3	No. 4	No. 5	No. 6	No. 7	No. 8	No. 9	No. 10	Avg.	Avg.
115	.018	.008	.013	.010	.010	.008	.010	.018	.008	.008	.011	.....
150	.189	.188	.185	.158	.172	.142	.158	.225	.194	.149	.176	.....
170	.208	.222	.194	.195	.192	.197	.198	.198	.175	.192	.197	.....
200	.895	.922	.866	.960	.879	.932	.873	.880	.884	.841	.893	.....
280	2.458	2.545	2.675	2.281	2.565	2.383	2.364	2.314	2.263	2.196	2.404	.....
Total	3.768	3.885	3.933	3.604	3.818	3.662	3.603	3.635	3.524	3.386	.....	3.682
Fines	8.792	7.475	6.235	6.121	7.935	8.301	7.460	8.746	9.848	7.888	7.880	.....
Total	12.560	11.360	10.168	9.725	11.753	11.963	11.063	12.381	13.372	11.274	.....	11.562
Min. T....	.040	.038	.042	.034	.035	.030	.022	.023	.036	.023	.032	.....

TABLE II. TEST NO. 2. WHITE SAND. 10 HOURS GRIND

MESH	No. 1	No. 2	No. 3	No. 4	No. 5	No. 6	No. 7	No. 8	No. 9	No. 10	Avg.	Avg.
115	.027	.029	.040	.025	.038	.029	.030	.032	.033	.....	.031	.....
150	.462	.471	.483	.470	.558	.581	.528	.570	.458	.....	.509	.....
170	.577	.609	.560	.599	.694	.543	.543	.460	.552	.....	.570	.....
200	2.092	2.028	2.249	2.139	2.011	2.033	2.124	2.280	2.181	.....	2.126	.....
280	5.798	5.842	5.564	6.418	6.355	5.522	5.620	5.695	5.925	.....	5.859	.....
Total	8.956	8.979	8.896	9.651	9.656	8.708	8.845	9.037	9.149	.....	.....	9.097
Fines	18.137	16.915	21.370	24.498	15.117	14.606	16.217	15.055	15.910	.....	17.536	.....
Total	27.093	25.894	30.266	34.149	24.773	23.314	25.062	24.092	25.059	.....	.....	26.633
Min. T.....	.062	.080	.074	.172	.098	.062	.045	.115	.065	.....	.085	.....

TABLE III. TEST NO. 3. WHITE SAND. 9 HOURS GRIND

MESH	No. 1	No. 2	No. 3	No. 4	No. 5	No. 6	No. 7	No. 8	No. 9	No. 10	Avg.	Avg.
115	.453	.435	.428	.460	.435	.469	.439	.457	.453	.447	.447	.....
150	2.206	2.164	2.185	2.230	2.170	2.046	2.191	2.195	2.224	2.248	2.185	.....
170	2.172	2.138	2.069	2.125	2.052	2.038	2.081	2.170	2.105	2.109	2.105	.....
200	5.493	5.850	5.662	5.612	5.765	5.811	6.037	6.041	6.350	5.600	5.832	.....
280	12.390	12.745	12.064	12.854	11.804	11.785	12.664	12.494	12.254	12.434	12.348	.....
Total	22.714	23.332	22.408	23.281	22.216	22.149	23.312	23.357	23.486	22.838	.....	22.908
Fines	18.542	18.880	17.780	18.460	18.776	17.582	16.242	17.381	17.030	16.425	17.709	.....
Total	41.256	42.212	40.188	41.741	40.992	39.731	39.554	40.738	40.516	39.263	.....	40.619
Min. T....	.120	.195	.115	.210	.100	.118	.130	.154	.153	.138	.143	.....

TABLE IV. TEST NO. 4. WHITE SAND. 8 HOURS GRIND

MESH	No. 1	No. 2	No. 3	No. 4	No. 5	No. 6	No. 7	No. 8	No. 9	No. 10	Avg.	Avg.
115	1.488	1.545	1.524	1.545	1.537	1.522	1.558	1.530	1.542	1.561	1.535	.....
150	4.425	4.538	4.698	4.580	4.647	4.725	4.615	4.652	4.690	4.645	4.621	.....
170	4.614	4.545	4.598	4.637	4.680	4.512	4.620	4.575	4.581	4.200	4.556	.....
200	10.450	10.389	10.330	10.122	10.374	10.522	10.418	10.575	10.045	10.660	10.388	.....
280	15.333	15.155	15.001	15.151	15.210	14.957	14.821	14.682	15.445	14.595	15.035	.....
Total	36.310	36.172	36.151	36.035	36.448	36.238	36.032	36.014	36.303	35.661	.....	36.136
Fines	14.040	16.562	19.270	16.337	14.775	16.745	17.310	15.255	15.731	17.031	16.306	.....
Total	50.350	52.734	55.421	52.372	51.223	52.983	53.342	51.269	51.034	52.692	.....	52.342
Min. T....	.182	.178	.140	.154	.170	.160	.135	.152	.138	.111	.152	.....

THE EFFECT OF CLAY ON THE FUSIBILITY OF BRISTOL GLAZES

BY E. C. HILL

In the series of terra cotta glazes I made last year, I found that the most fusible glazes were obtained with the lowest clay contents. The minimum clay for the different series was from .03 to .10 eq. In these glazes the RO contained BaO and SnO_2, in addition to CaO and ZnO, so that the amounts of CaO and ZnO were considerably lower than that for Bristol glazes. The minimum ratio of Al_2O_3 to SiO_2 in the different series were about 1 :7, which is a trifle higher than that for Bristol glazes. While these glazes were different in composition from Bristol glazes in these respects, the results regarding the clay content that would obtain the most fusible glazes, were so different from those of Prof. Purdy's results and conclusions concerning Bristol glazes that attention was called to these differences in the concluding sentence of my paper, "It is not evident why so much clay could be added to the Bristol glazes without decreasing the fusibility." This statement referred specifically to the amounts of clay that could be added to the glazes in Series I and II of the Bristol glazes reported in Vol. V of the *Transactions*. Prof. Purdy has justly inferred from this statement that I had neglected to take into consideration the fact that the CaO was higher in these glazes than in the terra cotta glazes, and that the action of the CaO on the clay would answer this implied question of facts. This statement should have been more explicit as to the amounts of clay that could be added without decreasing fusibility, rather than the reason why the clay could be added.

The intent of this statement was to promote a discussion on the effect of clay on both types of glazes. In the discussion which followed, Prof. Purdy justly stated that the compositions of the terra cotta glazes were so different that it was not a Bris-

tol glaze series, and that the effect of clay on the fusibility could not be compared.

Prof. Purdy has called my attention to another statement made in the discussion of the paper, "That where the CaO and ZnO are low as in this study, a high clay content decreases the fusibility, which is contrary to what I had learned about stoneware glazes." Prof. Purdy's objection to the above statement is not that "where ZnO and CaO are low, a high clay content decreases the fusibility," but to the statement that "this is contrary to what I had learned about stoneware glazes." The idea I wished to convey by this statement was that in the series of glazes I studied where the RO was more complex, making the CaO and ZnO contents considerably lower, the most fusible glazes were obtained with low clay contents, while in the porcelain glaze study by Prof. Purdy in Vol. XIII, he states the most fusible glazes were obtained with .50 and .55 eq. of Al_2O_3 which, in the case of the lower feldspar contents, would give a clay content of from .30 to .35 eq. The idea of the first statement is expressed further in the one which follows it, in which I said that "my experience has been that you get the brightest and most fusible glazes with the lower clay contents, but with high CaO and ZnO, the conditions are different."

After the discussion last year, my attention was directed to the articles by Prof. Purdy on Bristol glazes in Vol. V and porcelain glazes in Vol. XIII, regarding the effect of clay on the fusibility. The evidence in Vol. V is shown by the formulas of the most fusible glazes, in which the largest amount of clay could be added to increase the fusibility.

The Vol. V glazes referred to are given in Table I.

TABLE I. GLAZE COMPOSITIONS

No.	K_2O	CaO	ZnO	Al_2O_3	SiO_2	CLAY	FLINT	FUSING TEMPERATURE
IF	.20	.40	.40	.32	1.76	.12	.32	1030°C.
IG	.20	.40	.40	.36	1.98	.16	.46	1075°C.
IH	.20	.40	.40	.40	2.20	.20	.60	1075°C.
IIF	.25	.35	.40	.40	2.20	.15	.40	1120°C.
IIG	.25	.35	.40	.45	2.47	.20	.57	1115°C.
IIH	.25	.35	.40	.50	2.75	.25	.75	1120°C.
IIIC	.30	.30	.40	.39	2.14	.09	.16	1075°C.
IIID	.30	.30	.40	.42	2.31	.12	.27	1045°C.
IIIE	.30	.30	.40	.45	2.47	.15	.37	1055°C.
VIB	.40	.20	.40	.48	2.64	.08	.08	1035°C.

Referring to these formulas, it will be seen that in Series I with .20 eq. feldspar, the glazes IG and IH were equally fusible and contained .16 and .20 eq. clay. The ZnO is constant in all series, and the feldspar increases at the expense of CaO. In Series II, the most fusible glaze contained .20 eq. clay and was more fusible than the one with .15 eq. In Series III, the most fusible glaze contained .12 eq. The amounts of clay that gave the most fusible glaze decreased as the feldspar increased at the expense of CaO, until with .4 feldspar and .2 CaO, the glaze with the first addition of clay, .08 eq. was the most fusible one of the entire study.

In Vol. XIII, Porcelain Glazes, the limits of compositions are:

$$\left.\begin{matrix} .0\text{-}.8\ CaO \\ .0\text{-}.8\ ZnO \\ .2\text{-}.5\ K_2O \end{matrix}\right\} \quad .50\text{-}.80\ Al_2O_3 \ \Big\} 3.0\text{-}4.8\ SiO_2.$$

The ratio of Al_2O_3 to SiO_2 in all glazes is 1:6.

The effect of the clay on fusibility is summed up in the two statements as follows (pages 579-581).

"(1) Comparing the extremes of the series, it is noted that within any group, increase of clay decreases the fusibility of the mixtures. In Group I and II (.20 and .25 eq. feldspar) we probably started (according to Purdy's curves p. 158, Vol. V) with the most fusible mixtures and added clay to them, while

in other groups, we started with mixtures which had already been made less fusible by additions of clay, hence we should expect to have increased refractoriness. A **decrease** of clay in Group I from .3 eq. to .0 eq. would no doubt have **likewise decreased** the fusibility of the glazes of this group."

"(2) From the curves of Purdy (Vol. V) it is readily seen why, in Series C and D (CaO:ZnO as 1:1 and 1:3) each increase of clay above the minimum used in any one of the groups in this study should have increased the refractoriness of glazes. In members 1 and 2 (.50 and .55 Al_2O_3) of Series C (CaO:ZnO as 1:1) Groups I and II (.20 and .25 eq. feldspar) we no doubt have the most fusible mixtures that can be obtained with these RO's. Seger found that with .25 K_2O and with ratio of Al_2O_3 to SiO_2 of about 1:6 (the same as ours) he obtained maximum fusibility with .5 eq. Al_2O_3 and found that either less or more Al_2O_3 caused an increase in refractoriness. This mixture of Seger's is about the same as our II A1 (.75 CaO .25 K_2O .50 Al_2O_3 3.0 SiO_2).

These statements assert that considerable more clay could be added for maximum fusibility than was used in the Bristol glaze study in Vol. V.

The second statement asserts that, as far as the glazes with .20 eq. feldspar are concerned, that the most fusible glazes that can be obtained with the same RO as that in Series I, Vol. V, is with .30 and .35 eq. clay. The most fusible glazes in Vol. V were with .16 and .20 eq. clay, but .20 eq. clay was the limit of the series.

Regarding the effect of ZnO and clay on gloss, under general conclusions on the cone 7 burn, we find the following (p. 559): "When ZnO is .4 eq. or more, increase of clay from .3 to .6 eq. appears to have but slight effect on the gloss of the glaze. This is more pronounced as the feldspar increases, due, perhaps, to the total clay content being less and less as the feldspar increases." When ZnO is .4 eq. or more, the CaO content ranges from .4 eq. to 0. As far as gloss is concerned, it must be inferred from this statement that ZnO combines with clay as readily, or even more readily, than does CaO.

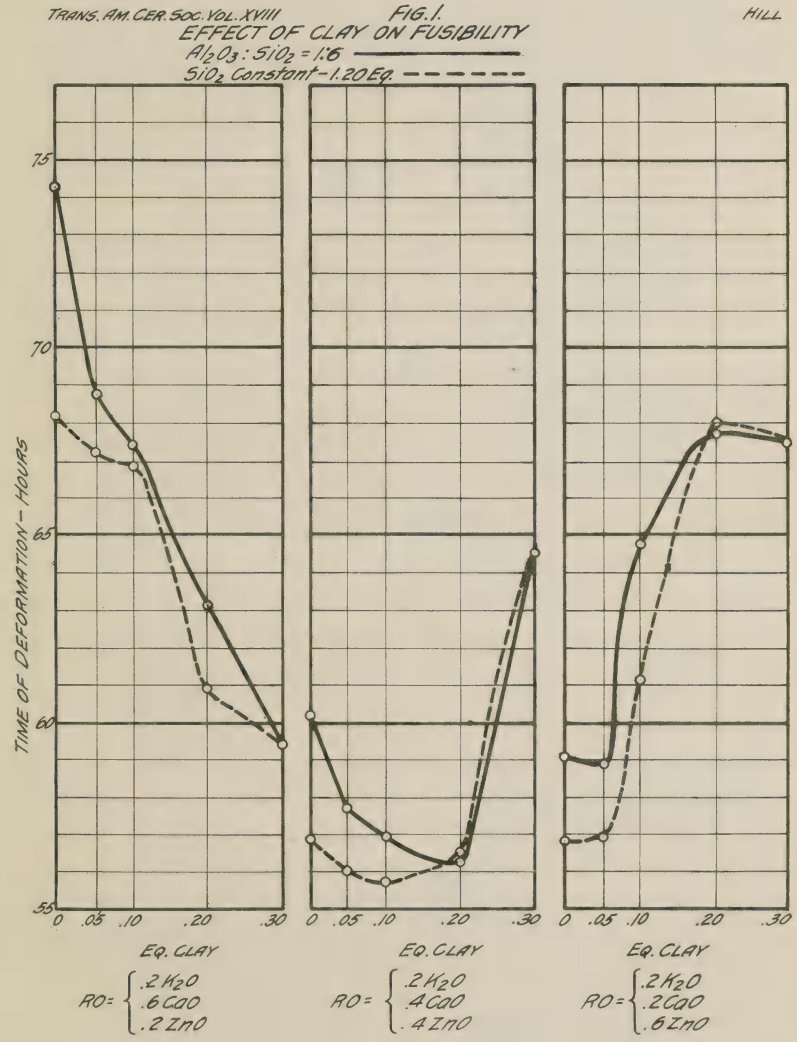
Regarding the effect of CaO and ZnO variations on gloss under the same heading as above, we find (p. 560) : " With low feldspar (less than .4 eq.) maximum gloss is obtained when the ratio of CaO to ZnO is less than 1 :1, while with higher feldspar, the ratio of CaO to ZnO must be higher, that is, at least 3 :1." From this, it must be inferred that for the low feldspar glazes, that ZnO is more active in producing gloss than CaO.

The evidence and statements cited left me somewhat in doubt as to the effect of ZnO and CaO on the clay, as regards fusibility and the amounts of clay that was required for the different CaO-ZnO variations to give a fusible glaze. This led to the series of glazes I reported in the discussion I wrote after the meeting last year. This series was planned to cover additions of clay between that used in the Vol. V study with .2 eq. feldspar and the Vol. XIII study; to study the effect of CaO-ZnO variations with the different clay and flint contents, and the effect of increased SiO_2. While it is well known that CaO reacts with both clay and flint, and that ZnO reacts with flint, I had not been able to find any data on the effect of ZnO on clay, except the statement cited above.

The results of this series are shown by the curves (Fig. 1) plotted from the data obtained.

$Al_2O_3 : SiO_2 = 1 : 6$. (1) CaO : ZnO : : 3 : 1. Any addition of clay up to at least .3 eq. makes the glaze considerably more fusible. This agrees completely with the statements of Prof. Purdy cited above.

(2) CaO : ZnO : : 1 : 1. This is the RO to which Prof. Purdy added the largest amounts of clay, .16 and .20 eq., and stated that the maximum fusibility was not reached. Clay and flint are added here to maintain an $Al_2O_3 : SiO_2$ ratio of 1 :6, while in Vol. V the ratio was 1 : 5.5. This curve shows that clay increases the fusibility up to .20 eq. which gives the most fusible mixture with $Al_2O_3 : SiO_2$, 1 : 6 but further additions rapidly decrease the fusibility. The glaze containing .30 eq. clay is more refractory than any containing less clay, even when the glaze contains no clay at all. This mixture is the one which Prof. Purdy says is the most fusible mixture that can be obtained


TRANS. AM. CER. SOC. VOL. XVIII
FIG. 1.
EFFECT OF CLAY ON FUSIBILITY
$Al_2O_3 : SiO_2 = 1:6$
SiO_2 Constant – 1.20 Eq.
HILL
TIME OF DEFORMATION – HOURS
75
70
65
60
55
0 .05 .10 .20 .30
EQ. CLAY
RO = .2 K_2O .6 CaO .2 ZnO
RO = .2 K_2O .4 CaO .4 ZnO
RO = .2 K_2O .2 CaO .6 ZnO

with these RO's, and to strengthen this assertion, he cites the glaze of Seger mentioned above.

(3) CaO : ZnO : : 1 : 3. The glaze with .05 clay is only a trifle more fusible than the one with no clay, and any addition of clay above .05 equivalents makes the glazes more refractory.

SiO_2 Constant, 1.20 eq. Flint. With SiO_2 constant in the amount to give the glaze with .30 eq. clay an Al_2O_3-SiO_2 ratio of 1 : 6, the clay was reduced as shown by dotted curve in Fig. 1. With CaO : ZnO as 3 : 1, the dotted curve follows the solid curve, except that for equal amounts of clay the glazes are somewhat more fusible. With CaO : ZnO as 1 : 1, the most fusible glaze has .10 eq. clay and the next in order of fusibility contains .05 eq. These glazes are more fusible than the most fusible one with the Al_2O_3 : SiO_2 as 1 : 6, which had .20 eq. clay. With CaO : ZnO as 1 : 3, the glazes with 0 and .05 eq. clay are the most fusible.

These results may have been more definite regarding the effect of clay on the various CaO-ZnO ratios if I had increased the clay in increments of .05 eq. between .10 and .30 eq. Seger found that with the same feldspar content as used here, and with .8 CaO, the most fusible glaze contained .3 eq. clay. Since curve III shows that ZnO does not combine readily with clay, as does CaO, we should expect that, when ZnO is added to a glaze it must contain less clay to get equal fusibility. Had the clay been increased in increments of .05, we might have found the most fusible glaze with .25 eq. instead of .30 eq. We might also have found in Curve II, that .15 eq. clay would give as fusible a glaze as .20 eq., since with this RO and a ratio of Al_2O_3 to SiO_2 as 1 : 5.5, Prof. Purdy found that glaxes with .16 and .20 eq. were equally fusible.

Conclusion. If the proportions of the various ingredients to give the most fusible glaze were known, these proportions should also give the longest heat range and should be generally free from crazing[1]. It has been shown, within the limits here studied, that the most fusible glazes were obtained with equal equivalents of CaO and ZnO and that an increase of either resulting in a ratio of 3 : 1 decreased the fusibility. The content

[1] Bristol Glazes, Purdy, *Vol. XIII, Trans. Amer. Cer. Soc.*

of clay to give the most fusible mixture with an Al_2O_3-SiO_2 ratio of 1:6 was .2 eq. Prof. Purdy fouund in Vol. V that with the same RO and with an Al_2O_3-SiO_2 ratio of 1:5.5 the most fusible glazes were with .16 and .20 eq. clay. An increase of SiO_2 resulting in an Al_2O_3-SiO_2 ratio of more than 1:6 increases the fusibility. The most fusible glazes of these series were those with comparatively high SiO_2 contents and containing .05 and .10 eq. clay, so that with the higher SiO_2 contents, the clay content must be considerably lower to secure the most fusible glazes. Having determined these relations, it gives a working basis for the development of a low-fusing terra-cotta glaze, by the addition and substitution of such components necessary to secure the desired properties.

Discussion by Author. After Prof. Purdy had read my discussion of the data just given, he prepared a discussion covering this, and my discussion of my paper last year. He has asked me to incorporate into this paper his discussion and to present both sides of the discussion of the effect of clay on fusibility on stoneware, porcelain and terra cotta glazes.

Why does clay increase the fusibility of stoneware, terra cotta, and porcelain glazes?

Prof. Purdy was surprised at the statement in my paper last year "it is not evident why so much clay could be added to the Vol. V glazes without decreasing the fusibility." This statement I have referred to before in this paper as questioning the amounts of clay, rather than the reason why the clay could be added.

As evidence of the amounts of clay that can be added without decreasing the fusibility, Prof. Purdy has given a number of references as to this fact in the *Transactions*. In addition to his own work in Vol. IV, V, XIII and XIV, he has given me the following:

Vol. VIII, p. 168, Fluxing Effect of Clay on Cone Mixtures Containing Little or No Clay, by Coulter. In this paper Coulter found that clay added to cone 2 increased the fusibility.

Vol. XV, Study of some Calcareous and Magnesian Slags by Bleininger, Brown and Kinnison. With the same iron con-

tent, the Al_2O_3 content has been raised nearly .4 eq. before decided refractoriness was obtained.

Vol. XVI, p. 454. Deformation Temperatures of Some Porcelain Glazes by Stull and Howatt. This study makes plain that clay must be added in comparatively large equivalents before greatest fusibility is obtained with mixtures having an R O of .30 K_2O and .70 CaO. The diagram with iso-deformation curves shows the effect of SiO_2 together with clay on fusibility. They show that irrespective of the SiO_2 contents, the clay must be present in .3 equivalents in order to have maximum fusibility.

Vol. XVII. Study of the Most Fusible Mixtures of K_2O, CaO, Al_2O_3, SiO_2, by Hanna. This contribution contains observations and conclusions that are surprising. They, however, like other references here cited, show plainly why clay is necessary to effect greatest fusibility, and in this case, even with a feldspar content of .6 eq.

American Journal of Science, Jan. 1915, Shepherd and Wright, have shown that their eutectic No. 2 is the most fusible one. Segers cone 4, therefore, is almost an exact mixture of feldspar and the most fusible eutectic of $CaO\text{-}Al_2O_3\text{-}SiO_2$ mixtures.

The evidence of these references cited above is very conclusive both as to why clay causes increased fusibility, and as to the large amounts of clay that can be added without decreasing the fusibility. There is no question as to this.

It is to be noted, however, that nearly all of the mixtures to which these references refer, do not contain ZnO, and in every case where Al_2O_3 increased the fusibility, the mixtures were high in CaO. The mixtures that did contain ZnO were those mentioned in the articles by Prof. Purdy.

The direct evidence of the effect of clay on fusibility is given in the Vol. V glazes already referred to. Referring to the glazes of Series I and II, to which my statement questioning the amounts of clay that could be added without decreasing the fusibility specifically referred to, it will be seen that Prof. Purdy started with a mixture of feldspar, whiting and zinc oxide, to

which he added clay, and enough flint to maintain a ratio of Al_2O_3 to SiO_2 of 1 : 5.5. The amounts of both added are given in the table with the formulas of these glazes. In Vol. V, Prof. Purdy states that the increase in fusibility, shown by the fusing temperature, is due to clay, and he has reaffirmed this in his discussion. There is no question at all but that SiO_2 added alone would have greatly increased the fusibility, so that the fusing temperature of these glazes would have been higher had clay alone been added.

Whether or not, the same amounts of clay could be added alone and still increase the fusibility, has not been determined with the RO's under discussion, or with any glazes containing ZnO. I have shown that with a content of SiO_2 to give the glaze with .30 eq. clay a ratio of Al_2O_3 to SiO_2 of 1 : 6, that is, 1.2 eq., that clay increased the fusibility only up to .10 eq. and that the glaze with .05 eq. was slightly more fusible than that with .20 eq. The answer to this question regarding glazes without ZnO, is found in the data on porcelain glazes by Stull and Howatt in Vol. XVI. Prof. Purdy has taken evidence from this deformation study to show the effect of clay. His statement is that the data shows "that irrespective of the SiO_2 content, the clay must be present in .3 eq. in order to have maximum fusibility." An examination of the table of deformation temperatures shows that this is entirely true. At the same time, this data shows that with the lowest SiO_2 content in this study, 2.0 eq., an increase of clay up to .3 eq. did not increase the fusibility. The deformation temperatures are the same for .10 eq. clay, the lowest in the study, as for .30 eq. clay which gave the most fusible glaze of all the Al_2O_3-SiO_2 variations. With each increase of SiO_2, however, up to that giving the most fusible mixture, the effect of clay in increasing the fusibility was more pronounced and had its greatest effect with 4.0 eq. SiO_2 which gave the most fusible mixture.

If this evidence can be applied directly to the point under discussion, Prof. Purdy could not have added the amount of clay alone to the Vol. V glazes and get increased fusibility. The fusibility would remain the same. At the same time, I am unable

to justify my statement questioning the amounts of clay that could be added to the Vol. V glazes, because it reads "that it is not evident why so much clay could be added without decreasing the fusibility."

[2] Porcelain, Stull, *Vol. XV, Trans. Amer. Cer. Soc.*
[3] Porcelain, Stull, *Vol. XV, Trans. Amer. Cer. Soc.*

NOTE ON THE VOLUME CHANGES OF SILICA BRICK MIXTURES.[1]

BY A. V. BLEININGER AND D. W. ROSS, PITTSBURGH, PA.

The manufacture of silica brick, apparently a very simple process, offers a series of interesting and important problems. The behavior of different brands of these bricks in the furnace is not the same, nor even is this so of bricks of the same make. There are variations with reference to volume changes subsequent to installation in the furnace and differences in regard to the resistance to sudden heating and cooling. It is evident that the inherent structure and the composition of the rock, the temperature and the duration of burning are important factors.

A study of silica brick has been undertaken by the Bureau of Standards of which this contribution is a part. The present study concerns itself with the volume changes occurring in silica brick during burning. The magnitude of the permanent expansion is usually considered the criterion of proper firing and amounts to from $\frac{3}{8}$ to $\frac{7}{16}$ of an inch per linear foot over the mold measurements. Assuming the complete transformation of quartz to cristobalite, a theoretical volume change of the substance of the silica brick of 13.4 percent is computed. Usually, however, the transformation is not complete. Upon longer continued heating practically all of the quartz is transformed, and, especially in the presence of fluxes, tridymite appears as the stable phase which also causes an additional though slight increase in volume.

The volume changes may, however, be very much larger. Thus Cramer[2] obtained as great an increase as 23.8 percent. This expansion, however, is not that of the substance itself but of the quartzite rock or of the brick as a whole.

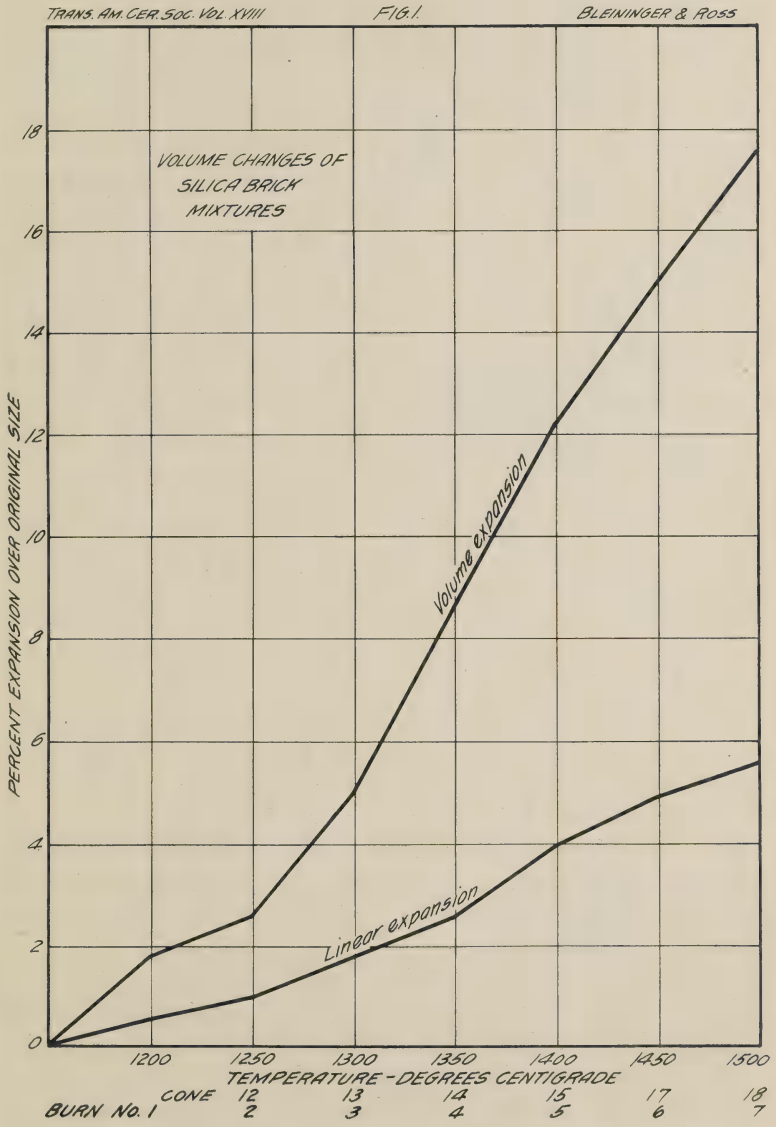
[1] By permission of the Director, Bureau of Standards.
[2] Cramer and Hecht, *Handbuch der gesammten Thonwaaren-industrie*, p. 893-97.

According to their structure and especially according to whether the crystals have suffered deformation in the folding process to which quartzite strata have often been subjected, the quartz grains tend to fissure and to break down to dust. This fine grained material, according to P. J. Holmquist[3], transforms more readily to the amorphous form and to tridymite than do the coarser grains. Cramer comes to the conclusion that quartzite rocks showing the greatest expansion during the first firing, cone 16-17, and comparatively small increases during subsequent firings, are most suitable for the manufacture of silica brick. This worker found that a certain pure quartzite showed a volume increase of 6.8 percent after the first and 18.5 percent after the sixth burn. Another specimen increased in volume 1.6 percent after the first and 11.5 percent after the sixth firing. On the other hand, another, evidently more impure material, was found to expand 13.9 percent (by volume) after the first and only 13.6 percent after the sixth heating. The latter type, therefore, appears to be more suitable for manufacturing purposes. The effect of the impurities, both those originally contained in the rock and those added in the form of lime, is to hasten the transformation to cristobalite, which means a lower subsequent expansion upon reheating.

Kenneth Seaver[4], in reburning commercial silica brick, found upon the third firing an increase in the cristobalite content of from about 77 to 84 percent. The temperature employed was approximately 1540° C. It would seem, therefore, that present commercial practice carries the conversion to cristobalite as closely to completion as is practicable.

In carrying out the test under discussion a prepared silica brick mixture was obtained through the courtesy of the Harbison-Walker Refractories Co., Pittsburgh, Pa., and made up into two inch cubes. The quartzite rock was that from the Tyrone district, Pennsylvania. These were fired to a series of temperatures from 1200 to 1500° C. The rate of heating was about 20° C. per hour. The maximum temperature was held for approximately four hours. The original and subsequent volume

[3] *Geol. Foeren Foerhandl* Vol. 83, No. 4 (1911).
[4] *Bull. American Institute of Mining Engineers* 1915.


TRANS. AM. CER. SOC. VOL. XVIII
FIG. 1.
BLEININGER & ROSS
VOLUME CHANGES OF SILICA BRICK MIXTURES
PERCENT EXPANSION OVER ORIGINAL SIZE
0
2
4
6
8
10
12
14
16
18
Volume expansion
Linear expansion
1200
1250
1300
1350
1400
1450
1500
TEMPERATURE - DEGREES CENTIGRADE
CONE
12
13
14
15
17
18
BURN No. 1
2
3
4
5
6
7

of the cubes were measured by means of the Seger voluminometer and the linear expansion was likewise obtained.

These measurements are shown graphically in the accompanying diagram. It appears from these data that expansion takes place at as low a temperature as 1200°C., although at this point practically no inversion to cristobalite is observed. At 1250° the first indication of cristobalite appears. Between these two temperatures a distinct lag is shown in the expansion curve. Beginning with 1300° the expansion appears to be quite constant, showing no acceleration or retardation as the temperature is raised. This steady increase in volume is coincident with, but not proportional to, the formation of cristobalite. It would seem from the form of the curve that a rather sudden termination is to be expected somewhere beyond 1500°C., which point was not reached however. It would incidate the most desirable burning temperature.

The amount of cristobalite was determined in the silica brick specimens at 1350°C. by sketching the fields on very thin tracing paper with the aid of a camera lucida. The paper was then weighed and the outlines of the grains cut out, whereupon the paper was reweighed. The amount of the crystalline phase in question was then readily computed. The accuracy of the method is probably $\pm$ 5 percent.

At 1300° the content of cristobalite did not exceed 5 percent, no tridymite being noted. A decided increase in this constituent was observed at 1350° when 57.13 percent was found, the remaining substance being quartz and calcium silicate. At 1400° this amount increased to 72.17 percent, at 1450° to 81.26 and at 1500° to 90 percent. But a small amount of tridymite was observed in all of the sections. It is apparent from these values that the volume expansion is not proportional to the cristobalite content alone since the curve is of practically constant slope even though a large amount of transformation took place between 1300° and 1350°. It is likewise evident that the linear expansion does not agree with the volume expansion. The former shows a dropping off at 1400° and again at 1450° and appears to approach a region of constancy. There is also lack of agreement between the linear and volume expansion. The latter point is

explained by the well-known fact that the expansion, like shrinkage, is not uniform in all directions. The linear changes in this work were measured only in one direction. The volume change seems to be somewhat greater in each case than the theoretical values computed from the linear measurements. Thus a linear expansion of 3 percent actually corresponds to a volume increase of 9.6 percent instead of 8.73 percent.

These measurements tend to show also that the minimum burning temperature of silica brick should be cone 18. It would seem, however, that the suggestion of Grzimails[5] to burn all silica brick till complete conversion to tridymite takes place, is practically not feasible, owing to the very heavy cost of carrying the temperature so high. The best criterion as to the best firing temperature would be that corresponding to the theoretical break in the expansion-temperature curve. By maintaining such a temperature, probably in the vicinity of cone 20, it might be possible to cut down the time of burning considerably.

The writers are indebted to Messrs. A. A. Klein and H. A. Brady for the petrographic determinations and measurements.

[5] *Stahl und Eisen*, 1911, No. 6.

THE EFFECT OF NON-PLASTIC MATERIALS ON THE SHRINKAGE AND MECHANICAL STRENGTH OF A NO. 3 FIRE CLAY BODY

BY W. C. MILLSOM, H. S. ROBERTSON AND C. C. TREISCHEL,

URBANA, ILL.

Realizing that the manufacturers of sewer-pipe are sometimes in a quandry when it becomes necessary to explain certain drying losses and certain failures of their product to stand high crushing stresses, we have made the following tests with the hope that they might prove of value in alleviating these difficulties. Of course we cannot hope to better the product of the man who has a shale which dries perfectly; but it is for the manufacturer who is forced to add a non-plastic material to his clay that this article is intended. Our object then, is to determine the effect, upon the drying and burning shrinkages and upon the crushing strength of the finished product, of varying the quantity and character of non-plastic materials added to a sewer-pipe clay.

The clay used in the tests was a No. 3 fire-clay of the Monmouth, Ill., vein, from Macomb, Illinois. This clay was chosen because of its availability in large quantities, rather than for its importance as a typical sewer-pipe clay. The grog used was obtained by grinding broken and imperfect pipe, since this is the source of supply in the commercial plant. The sand-rock was a round grained variety of rather good quality and also came from the Monmouth district. The calcined clay was the same No. 3 fire-clay which was used for the clay substance and was preheated to 600 degrees. The calcining was done in a downdraft gas kiln, known as kiln No. 6 at the University of Illinois kiln laboratory.

The different ingredients were first ground in a dry pan equipped with a 10 mesh screen. The body mixtures were then weighed out according to the following table and then ground in a wet-pan for fire minutes.

TABLE I. BODY MIXTURES[1]

Series A

NUMBERS	1	2	3	4	5	6
Clay	100	90	80	70	60	50
Grog	.....	10	20	30	40	50

Series B

Clay	90	80	70	60	50	.....
Sand	10	20	30	40	50	.....

Series C

Clay	80	70	60	50	40	.....
Grog	10	20	30	40	50	.....
Sand	10	10	10	10	10	.....

Series D

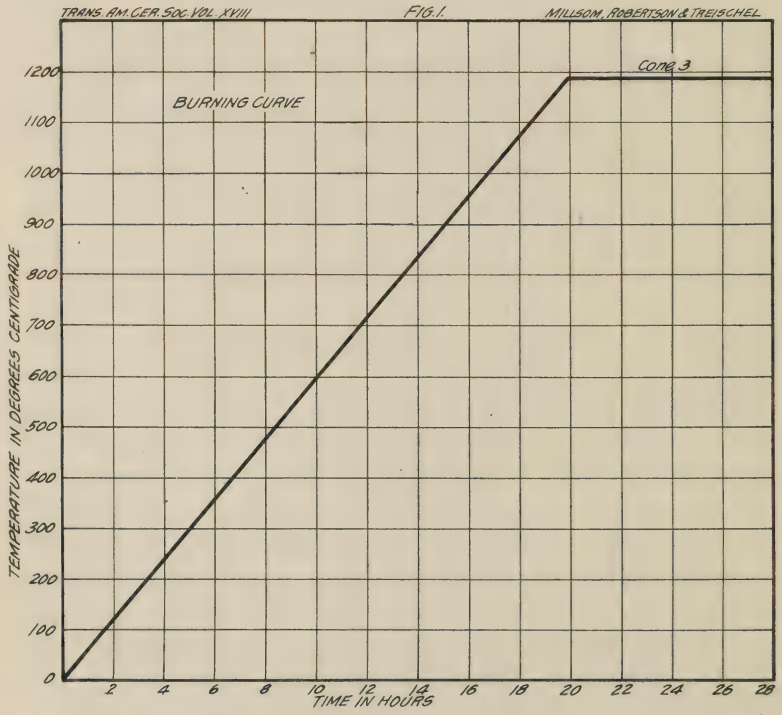
Clay	80	70	60	50	40	.....
Grog	10	20	30	40	50	.....
Calcined clay	10	10	10	10	10	.....

[1] Figures are parts by weight.

From the wet-pan the tempered bodies were transferred to a small auger machine and run out through a 2½ inch drain tile die. These tile were cut into 12 inch lengths. The tile were allowed to dry in the air for 24 hours and at the end of this time it was noticed that nearly all of Series A had cracked along the whole length of the tile where the cross bar of the die had cut the clay in front of the auger. This series was then re-tempered and run through the die again, and six pieces of the 2½ inch drain tile, twelve inches long were again cut off and allowed to dry. This difficulty did not then occur again, and after allowing the tile to dry for from two to three days, they were set in the kiln, ready for burning. The tile were set upright, two deep, on rings of moist clay run out through the same die. The rings were about two inches high.

At the same time that the tile were run out on the small auger machine, four brickettes, hand pressed on a small dry-press machine, were made and marked for determining drying shrinkage, and burning shrinkage. These brickettes were set in the kiln in saggers, and tightly sealed to keep out the salt gases.

The first burn was made by setting three tile of each body in the kiln in the manner stated above and placing at the same time the saggers containing the brickettes.



Burning Conditions. On account of the smallness of the tile, the water smoking period and oxidation period were not extended to any great length of time as can be seen by the curve. See Figure 1. The kiln used was a rectangular, down-draft, open-fire type of kiln, provided with two fire boxes. This is the test kiln known as Kiln No. 1, in the kiln laboratory of the University of Illinois. The fuel used was soft coal.

Method of Salt Glazing. In order to make the test approximate commercial conditions the ware was salt-glazed. The burning was continued until cone 3 went down, and the temperature was held at this point for three hours, a good thick bed of fire being obtained in this way. Salt was then applied at one of the fire boxes by scattering about ten pounds of salt over the fire with a shovel. This was repeated three times, salting first one fire box and then the other. The last salt was made by salting both fire boxes at once, closing the damper and letting the fire die out in this manner.

On account of the smallness of the kiln and the thinness of the fire on this burn, great difficulty was encountered by the temperature of the kiln dropping at the time of applying the salt, so that from one to two hours was required to raise the temperature up to the required point after each application of the salt.

Draw trials were made during the burn and at the highest point reached and the ware seemed to be vitrified, but, from the appearance of the tile when finished it was evident that these draws lay in a hotter part of the kiln than the rest of the ware as the drain tile themselves were not vitrified. The product of this kiln was then of little value for testing, so a second burn was made.

The setting and burning of the second kiln was the same as the first, with the exception that the temperature was raised about thirty degrees higher than on the first burn.

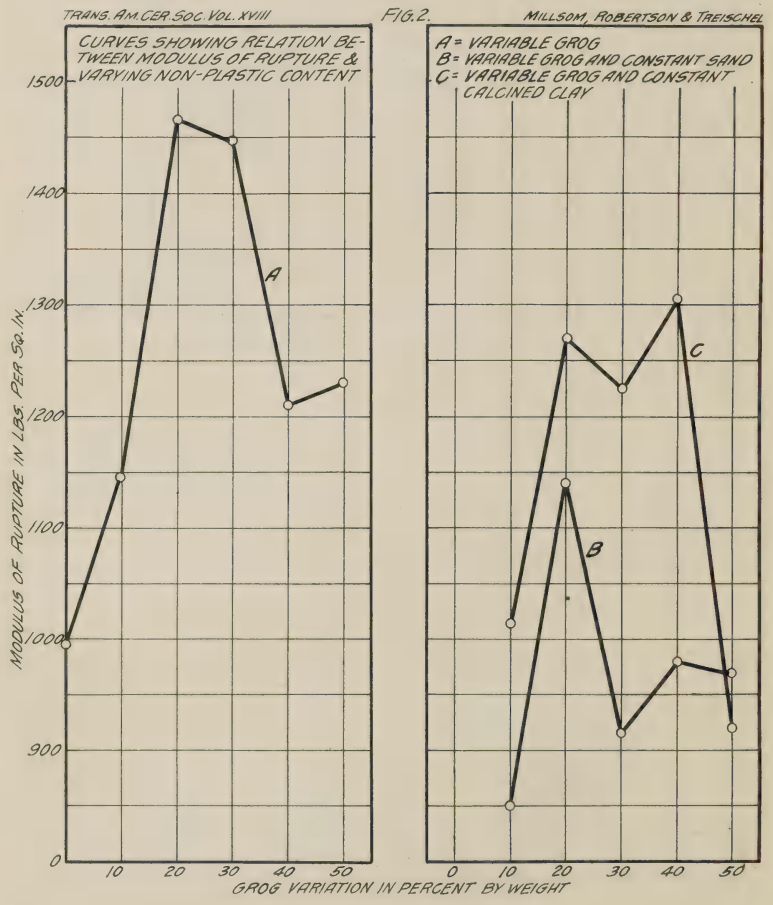
In this case the ware was completely vitrified and took a much better salt-glaze than the first, although not quite equal to a commercial glaze. The salt glazing of the tile was handicapped to a certain extent by inexperience. This was the first salt glazing attempted in this kiln or at the University of Illinois.

The ware from this kiln was tested for crushing strength.

Testing of Ware. The place of testing the ware for crushing strength was the Theoretical and Applied Mechanics Laboratory of the University of Illinois.

The machine used for the crushing of the ware was a 100,000 pound, Riehle testing machine in use at the laboratory.

The tile were laid horizontally on the machine, between two wooden blocks a little longer than the tile. Strips of leather were laid between the block of wood and the tile, in order to



take up as much as possible the irregularities on the outside of the tile, due to warping.

These irregularities in the tile, due to the warping, were the main sources of error in the testing. The errors were eliminated to a certain extent, however, by breaking three tile of

each body. The data from these three were then taken and the modulus of rupture calculated for each. The two results that checked the closest were then taken, and the average of these two recorded as the modulus of rupture of that body. While the warped tile may have caused uneaven bearing surface and therefore some error, yet we think that the comparative results as averaged above, give us reasonably accurate results to work on.

The data taken were: 1, the thickness of the walls, t; 2, the inside and outside diameter of the tile, which, when averaged gave us the mean diameter of the tile, d; 3, the length of the tile, l; and 4, the load necessary to crush the tile, P.

$$\text{Modulus of rupture then equals } \frac{.96\,Pd}{lt}$$

$$\text{The drying shrinkage in percent equals } \frac{\textit{wet length} - \textit{dry length } X\ 100}{\textit{wet length}}$$

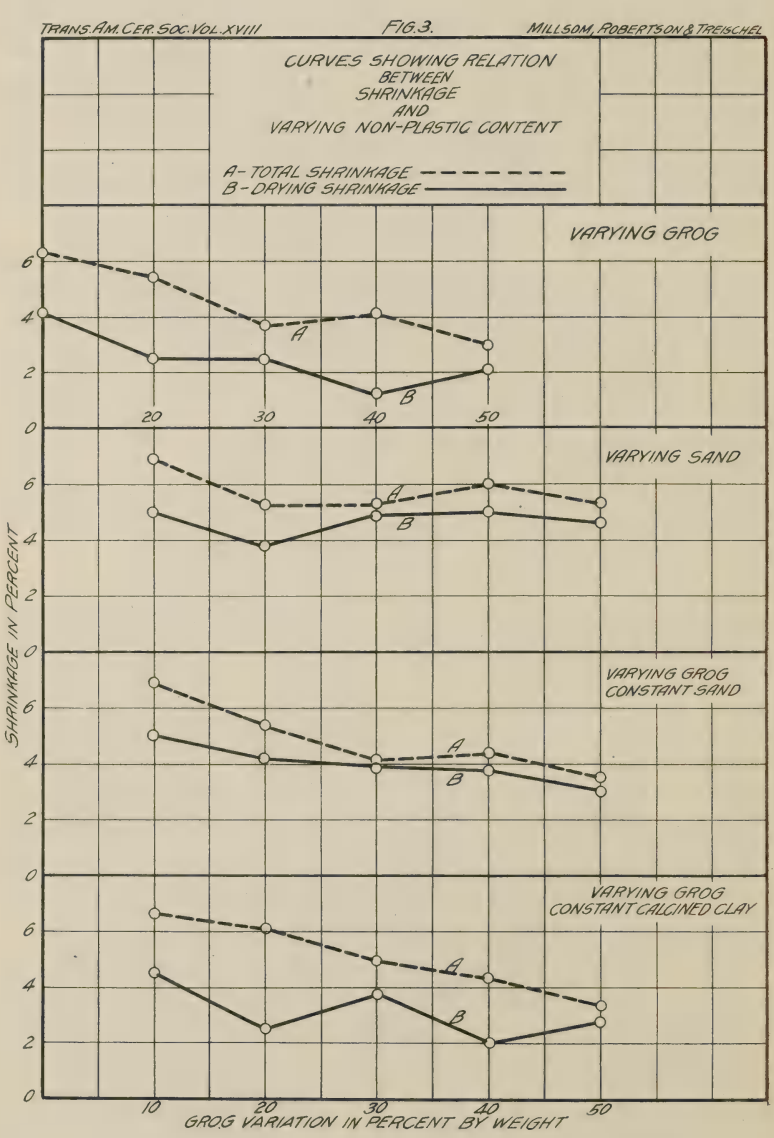
$$\text{The burning shrinkage in percent equals } \frac{\textit{wet length} - \textit{burned length } X\ 100}{\textit{wet length}}$$

Conclusions. 1. Due to an excess of silica all of the B series cracked, excepting B1 which contained only 10 percent sand. The probable reason for this is the polymorphus transformation which the free silica passes through in the cooling, from β to α quartz. This could also occur in commercial practice.

2. The curves show that the strongest body occurs with the use of grog, from about 20 to 40 percent.

3. In the series where calcined clay replaced sand, we have greater strength, so that we are safe in assuming that the sand does not add to the strength of the body.

4. From the shrinkage curves we see that the smallest shrinkage occurs in the grog series, while in the mixed series the grog and calcined clay gives the lowest shrinkage.


TRANS. AM. CER. SOC. VOL. XVIII
FIG. 3.
MILLSOM, ROBERTSON & TREISCHEL
CURVES SHOWING RELATION BETWEEN SHRINKAGE AND VARYING NON-PLASTIC CONTENT
A - TOTAL SHRINKAGE
B - DRYING SHRINKAGE
VARYING GROG
VARYING SAND
VARYING GROG CONSTANT SAND
VARYING GROG CONSTANT CALCINED CLAY
SHRINKAGE IN PERCENT
GROG VARIATION IN PERCENT BY WEIGHT

5. From these results, we think that we can safely say that as an ingredient in a sewer pipe body, sand is an undesirable element in so far as strength and shrinkage is concerned, and if the plant breakage is so low that there is an insufficiency of grog, calcined clay may be substituted for it. It might also be wise to substitute calcined clay for some of the sand if it can be done without affecting the quality of the salt glaze.

RESULTS OBTAINED IN FIRING SANITARY EARTHENWARE IN THE DRESSLER TUNNEL KILN

BY C. J. KIRK.

Last August, it was our great pleasure to receive a visit from a number of the members of this Society, who came to New Castle to see the first Dressler tunnel kiln that had been erected

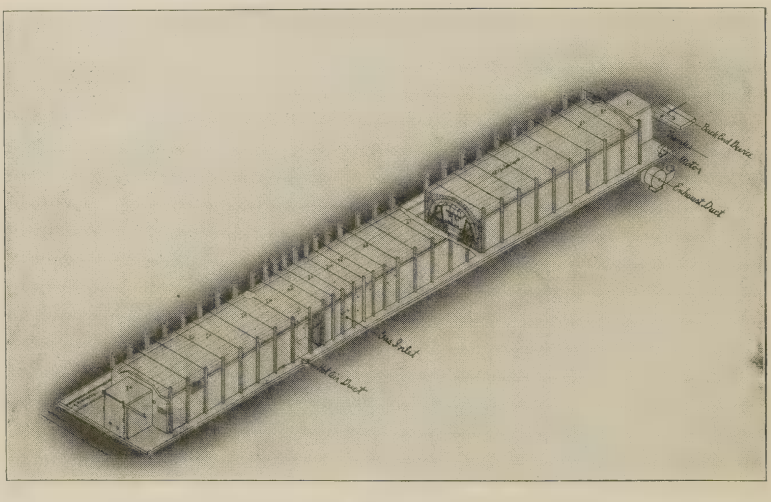


FIG. 1

in this country, in fact, the first one erected in any country for firing sanitary earthenware. A number of the members of the Society expressed a little skepticism as to the claims made for this kiln, and I thought you might be interested in hearing some of the results we have obtained in firing this particular kiln in the last four months. About the only claim made by the English company who furnished this kiln that was not carried out was, that we would not have any trouble with it, and that we could

start off and fire our ware without any loss. Unfortunately they never had fired any sanitary earthenware, and we did have trouble. We began to fire the kiln about the middle of September, using natural gas as fuel, and we started it by putting saggers and fire brick on our trucks and running them through. The manufacturer who furnished us our cars had not made them properly, they were not square on the ends, consequently when we pushed

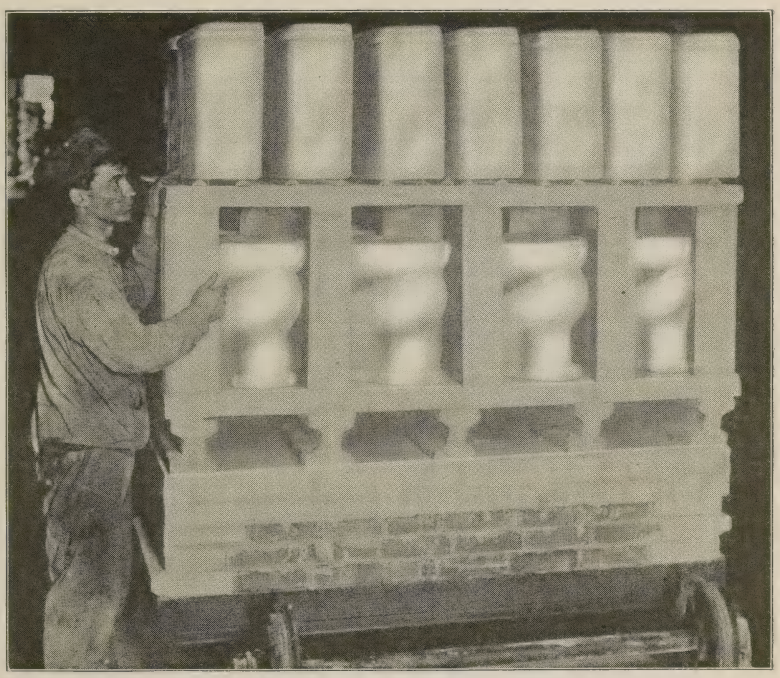


FIG. 2

one car in and then put in another to push the one ahead of it, they would not run evenly on the tracks, and were pushed all sorts of ways and developed a tremendous friction on the tracks and rollers on which they were running. That stripped the gears on our pushing device and finally we had to resort to 50 ton hydraulic jacks to push the cars through. That took some labor and time. We overcame this difficulty by chipping off one side and

putting a little metal bushing on the other side and truing the cars up. After we had made these changes, it is remarkable what a small amount of power is required to operate. We had four men on a hydraulic jack before we got these properly adjusted, it now takes one-twelfth of a horse power to push the cars through. Another trouble that developed was with the rollers. These rollers were placed with two in a span, and the spanners were put together with malleable connections which cracked in riveting them, and they broke down in the kiln, and we had to cool the kiln down to take them out. We overcame that by putting on wrought iron connections.

After overcoming some of these minor details, we began to do business. We have been running the kiln now for about three months continuously, and the claims made for the amount of fuel it would take to operate this kiln and for the labor have all been fulfilled and more than justified the expense and labor we have expended on it. We developed another trouble as the weather got cooler; in putting the ware into the kiln, we cracked about 25 percent of it in fire cracks, and in taking it out, we dunted another 25 percent. That was rather discouraging, but we built hot rooms around the end of the kiln and put our ware in these rooms, and the heat from the kiln heated it up so that we overcame that trouble. Fig. 1 shows a sectional view of the kiln; it is 245 feet long and about 20 feet wide, and the gas is applied at a place about two-thirds of the distance from the feeding end, and is carried in the chambers to the end of the kiln and withdrawn with a fan and delivered through an 18-in. pipe. We first put it out through the roof. As the weather got colder, we did not think it was necessary to waste all that heat, and took it through the slip house, and heated part of our shop. Figure 2 shows the tanks and the closets, that we fire, on the truck. The lower part of the car is built up of fire brick, and at the bottom it is made of steel angle plates and runs on rollers. The success of this kiln depends on the circulation of the hot air around the chambers. Figure 3 shows the hollow chambers. As the air is cooled by the ware, it drops down, goes around the fire chamber and is circulated over and through the goods; in this way it keeps moving and equalizing the temperature. We find on testing

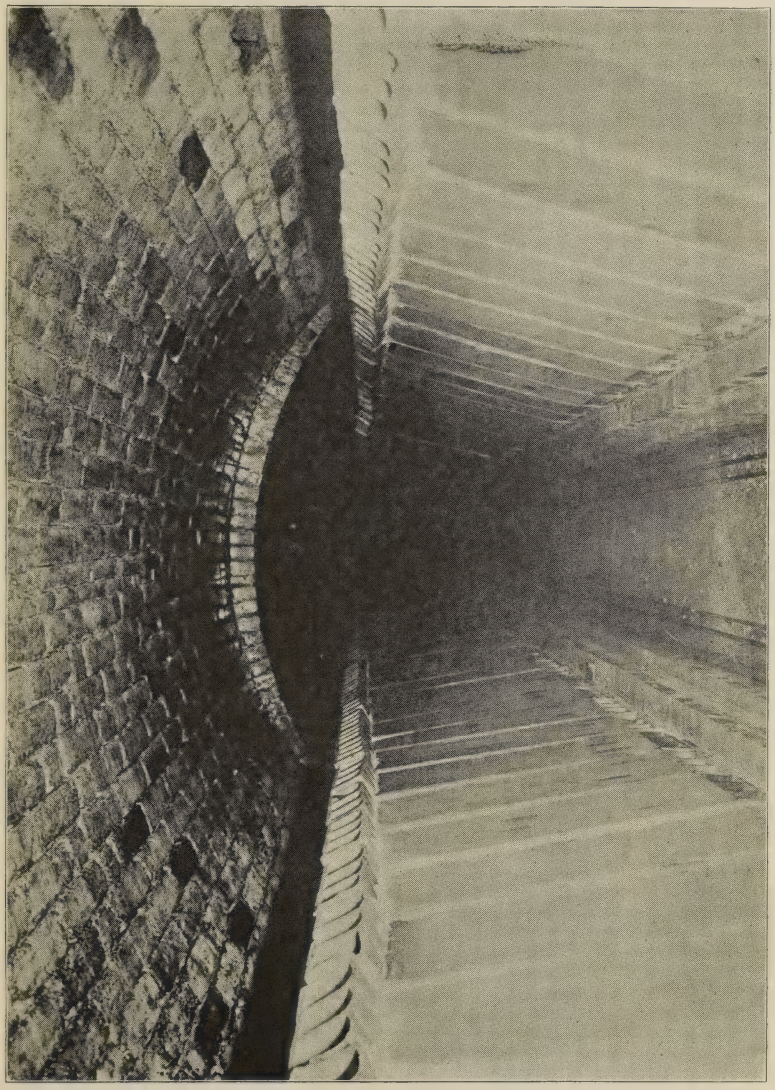


FIG. 3

it with cones, that there is not one cone difference between the top and the bottom the space in which we fire the ware, and this space is about six feet high and 5 feet wide. We found that this space was best adapted to our ware. Fig. 4 shows the pushing gear—the cars are introduced at this end, and pushed through; they come out at the opposite end and are pushed along the side of the kiln and unloaded. Then they continue down and are again loaded with ware. Figure 4 shows the rack and pinion gear that pushes the cars through. The cars travel at the rate of one inch per minute. Table I shows the result of our gas consumption and the heat records by a pyrometer for a week or eight days. On December 7th we used 29,000 cubic feet of gas in twenty-four hours, and the pyrometers are spaced about 20 feet apart through the kiln. Where we enter the temperature is about 450 degrees F., twenty feet further along it is 700 degrees, and the temperature increases until it is 1980 at the hot zone in the kiln. At that point we had No. 3 cone down and cone 4 bent. The next day we used thirty thousand cubic feet, and the heat advanced a little with cone 4 down. The next day we used twenty-four thousand cubic feet, and the heat went up to 2,000 degrees and the No. 4 cones were all down. Then it settled down, and we are using about twenty-five thousand cubic feet in 24 hours, and the temperature is carried fairly constant. We hold that temperature, and it does not vary 10 degrees in 24 hours, and we consider that it is very satisfactory for our ware. I would say that we were getting in the 24 hours, three hundred pieces of ware, tanks and bowls, the average weight is about 50 pounds each, making 15,000 pounds material put through the kilns in 24 hours. To fire that same amount of ware in an upright kiln would take from 250,000 to 300,000 feet of natural gas. That will give some idea of the economy of fuel in this kiln. Figure 4 shows our pushing device, a rack and gear pinion, and it is thrown out of gear with an automatic device. This rod is pushed back and the car run in place and pushed into the kiln, the door is closed and the cars are kept moving continuously. The length of the cars is five feet, six inches, and 40 cars are in the kiln at one time, twenty-eight of these from the charging end to the fire. The cones are put on the end of the truck, and

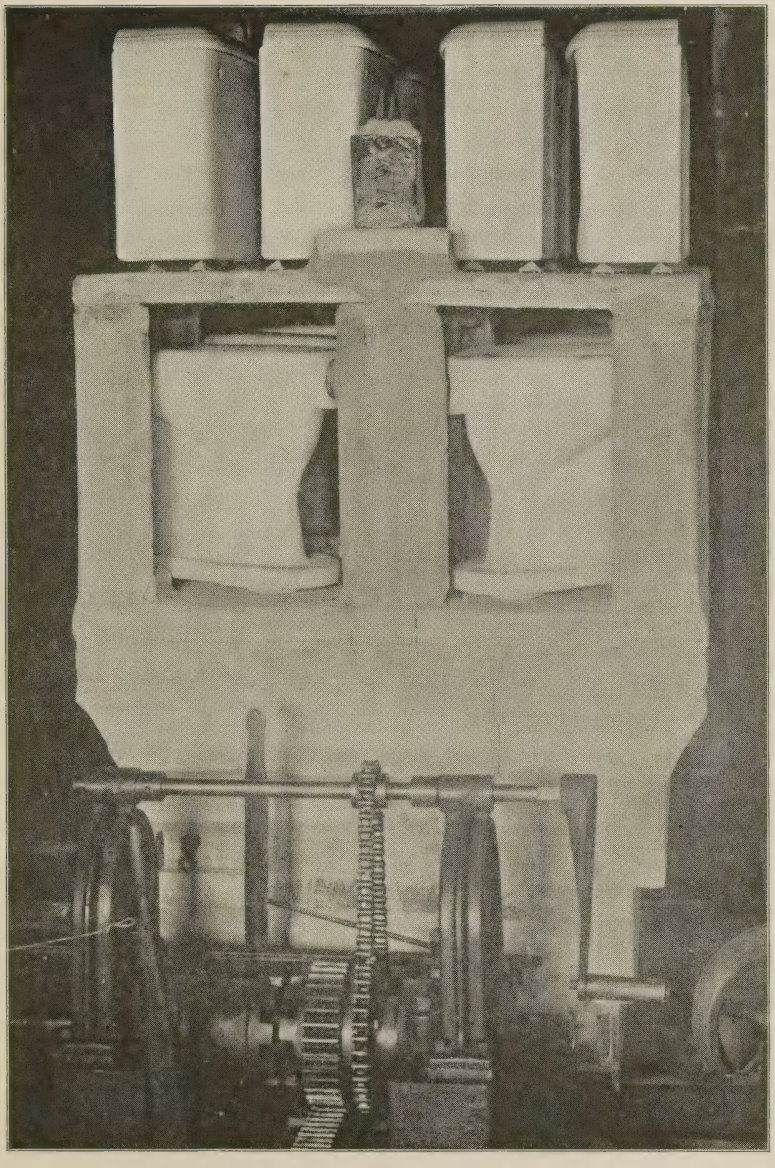


FIG. 4

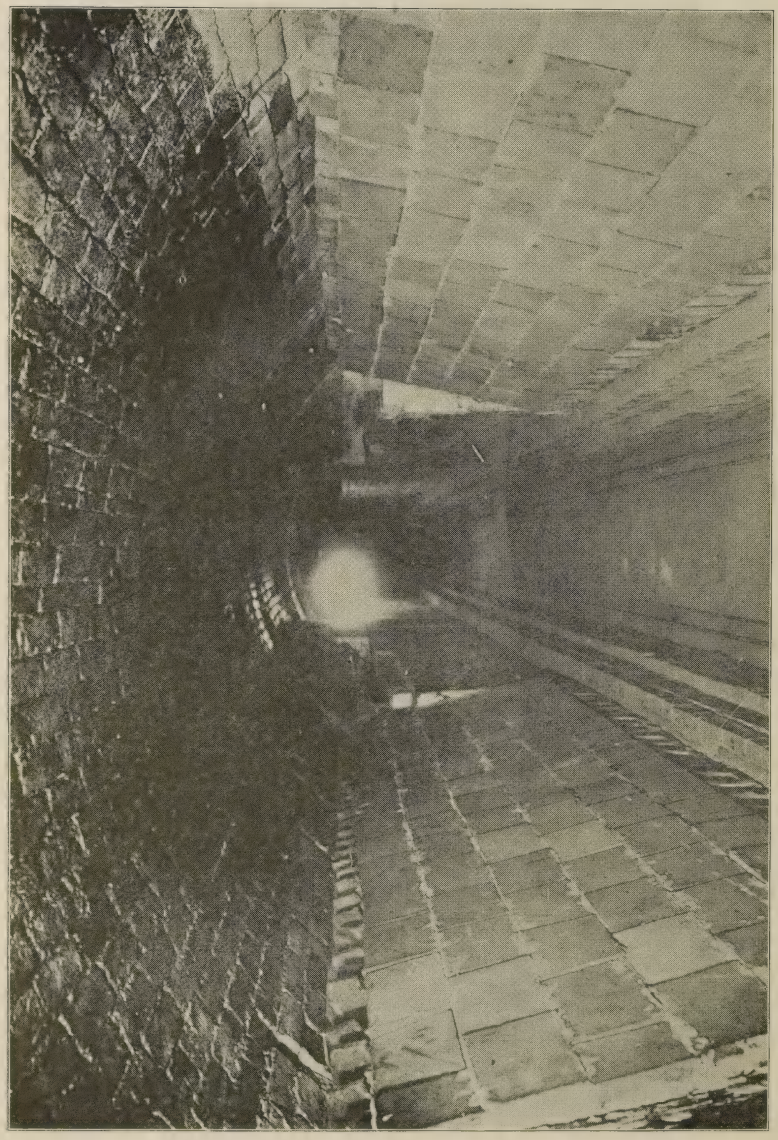


FIG. 5

they can be seen from the inspection holes. Figure 2 gives you a side view of the truck. We put eight closets and fourteen tanks on a truck. Figure 5 is a photograph taken of the combustion chambers—this is where the combustion takes place—these are fire brick benches, and this section of the chambers are made from carborundum and they are faced with fire brick. The openings in the top of the flue show where the heat is carried down and drawn back through the chambers to fire the ware coming in.

After four months run, there was practically no deformation on these chambers whatever, with the exception that in putting in the foundation, under the hottest part, the engineer had permitted the use of Portland cement grouting, and this grouting, under the intense heat, disintegrated and let these flues go down a little, and they bent over until they touched the sides of the kiln, and in expanding, they had been twisted and some of these parts opened up. They were not affected in any way by the heat, but were opened up where joined together and allowed some of the air from the kiln to pass into the flues, and it interferred with the vacuum that we keep there with a fan.

Figure 5 shows the fire clay chambers, and they are practically intact. They have not deteriorated in any way that we can see after a four month's use. About the 15th of January, we had some zero weather, and the Gas Company notified us that we would have to quit using natural gas. To prepare for this, we had erected a gas producer, and we started this up and began to fire the kiln with fuel gas. We found with natural gas, the heat was developed and carried along, as you see in that pyrometer record. With producer gas, the flame carried farther, it took longer for combustion.

Fig. 6 shows the end of the kiln where the cars are entered—these are cast iron pipes and the products of combustion are carried down in a pipe and go out through the fan. (See Fig. 7). The heat was so great at this end that we could not introduce our ware without cracking it. The producer gas carried the flame and heat farther along to this end of the kiln: in fact, these got red—with natural gas we could not see any color for thirty feet—but with producer gas, it ran the heat at this point up to about 800 degrees, and we had to add 20 feet to this end of the kiln, and

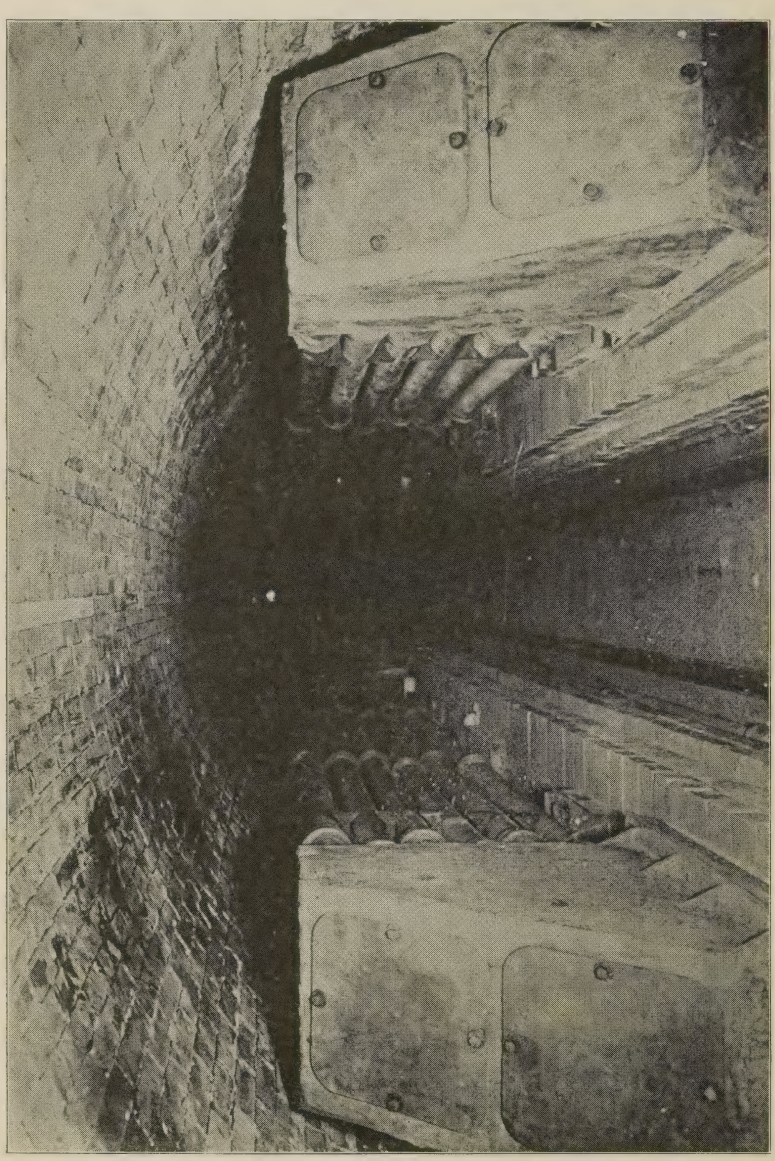


Fig. 6

we are just now completing that operation. Mr. Dressler, who is the patentee of the kiln, has returned from England. He is with us and we are ready to start up again with producer gas.

At the time this copy was gone over, on June 29th, the kiln had been in continuous operation for five months, part of the time on producer gas and part on natural gas, and our losses from broken ware are less than one percent.

We have never been able to make enough ware at our place to keep it going, to run it up to its full capacity. It runs twenty-four hours a day, and thirty days a month, and we have had to double our capacity, and we are still unable to keep up with it. It turns out more ware than we have been able to produce yet, but we hope to catch up to it some day.

In England they have been using this kiln now continuously for eighteen months and they shut it down because they thought they ought to look through the flues, and they found no deposits of any kind. The secret of the economy of fuel in this kiln is the fact that we take the hot air from the goods that are cooling and introduce it into the flue to fire the goods that are coming through, and we get a splendid result in that way — that air goes into the flue at probably 1900 degrees, and we only heat it up one hundred degrees with the fuel to fire the goods, and with producer gas, we get perfect combustion — in fact, it was too perfect, it carried the heat clear to the end of the flue and made it too hot.

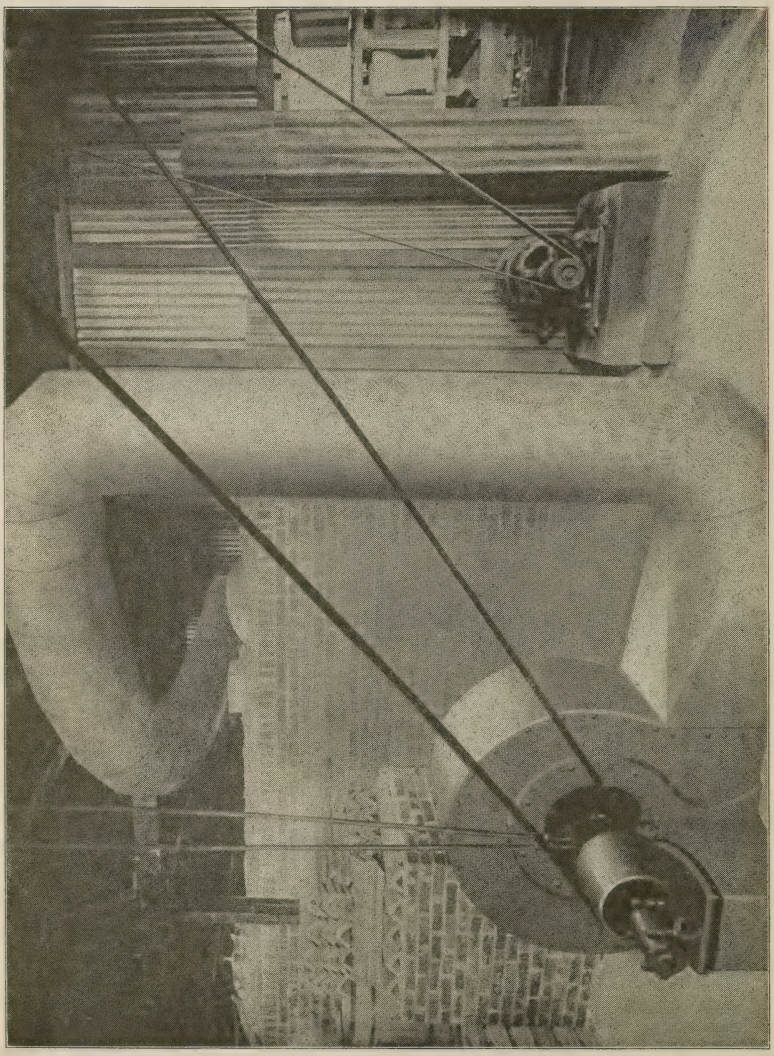


Fig. 7

TABLE I

DATE	GAS CONSUMPTION	PYROMETER NUMBERS															CONES 1-4
		1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	
	Cubic ft.																3 down
Dec. 7	29,000	450	700	890	1020	1270	1500	1740	1980	1960	1900	1560	1240	1100	920	460	4 bent
Dec. 8	31,000	460	770	890	1020	1280	1500	1760	1990	2000	1920	1650	1270	1160	950	460	3 down
Dec. 9	24,000	450	760	880	1020	1240	1450	1705	2000	2000	1940	1700	1280	1180	970	450	all down
Dec. 10	28,000	450	750	860	1000	1250	1480	1720	2030	2000	1980	1780	1360	1200	990	460	all down
Dec. 11	25,000	450	770	880	1020	1280	1520	1780	2060	2020	1980	1720	1340	1240	1000	480	all down
										2000	1960	1700	1380	1220	970	460	all down
Dec. 12	25,000	460	760	860	1020	1250	1480	1740	2040								3 down
Dec. 13	25,000	450	740	840	1000	1250	1460	1720	2040	1980	1920	1700	1360	1220	1000	480	4 bent
Dec. 14	25,000	460	760	860	1020	1270	1490	1740	2040	2000	1960	1760	1360	1240	1020	480	all down

NOTE — STONES IN GLASS: ANOTHER POSSIBLE CAUSE.[1]

BY ALEXANDER SILVERMAN

Numerous explanations have been advanced to account for the formation of stones in glass, but as far as the writer has been able to learn, nothing has been published concerning the influence of alkali chlorides.

Sodium and potassium chloride occur as impurities in raw materials, and in recent years the former has been added to many alabaster batches. Both vaporize at the high temperature of the glass furnace, and the vapor has a tendency to attack the clay of the pot roof. Some manufacturers use an excess of salt in alabaster batches. The excess floats on the surface of the glass exerts a powerful action on the pot.

Through corrosion by either the vapor or the liquid, the pot wall becomes pitted and small particles are loosened, entering the glass. The use of chlorides should be so regulated as to avoid the introduction of more than will dissolve perfectly in the glass.

[1]Contribution from the School of Chemistry, University of Pittsburgh.

THE MECHANICAL STRENGTH OF FIRE-CLAY BODIES AFTER REPEATED HEATING

BY F. A. KIRKPATRICK, URBANA, ILL.

INTRODUCTION

Very little work has been done to determine the properties of fire-clay bodies after repeated heating at high temperatures. Saggers, crucibles, glass pots, retorts, kiln and furnace parts, etc., are subject to continuous and repeated heating and in general deteriorate rapidly in use.

The need for investigations along this line is indicated by a number of articles in the *Transactions,* especially those by Simcoe in Volumes XI and XVII. The rate of loss of fire-clay saggers with repeated heating at cones 12 to 13 is indicated by one of Simcoe's experiments[1] as follows:

No. of Fire	*Good Saggers*
1	149
2	140
3	121
4	108
5	80
6	64
7	55
8	51
9	39
10	22
11	0

In this case a large loss occurred in the third and in each successive burn. Simcoe[2] also showed that in eleven New Jersey plants

[1] *Trans. Amer. Cer. Soc.*, Vol. XI, p. 222.
[2] *Trans. Amer. Cer. Soc., Vol.* XVII, p. 256.

making electrical porcelain, tile, and general ware, the average life of a sagger was from five to seven burns.

Sagger loss has been decreased in many cases by varying the kinds and amounts of clay, grog, and other materials and by greater care in manufacture. Better saggers are obtained by using several different bond clays than by using only one. Simcoe gives the following average composition of sagger mixes used in the eleven New Jersey plants:

	percent
Tight burning plastic clay	10
Open burning plastic clay	20
Tight burning sandy clay	17
Open burning sandly clay	7
Grog	46

The literature on the subject of fire-clay refractories is extensive, and a review will not be attempted at this time.

EXPERIMENTAL PART

In order to obtain some idea as to the course of the deterioration of fire-clay bodies after repeated heating, mixtures made up of 50 percent grog and 50 percent of a single clay (by weight) were burned repeatedly and after each burn the following properties were determined:

Percent porosity,
Percent absorption,
Apparent specific gravity,
True specific gravity,
Enclosed pore space,
Modulus of rupture.

Seven clays were used: 1, Illinois pot clay; 2, Ohio fire clay; 3, Kentucky pot clay; 4, New Jersey fire clay A; 5, New Jersey fire clay B; 6, St. Louis fire clay; 7, Tennessee ball clay No. 7. The grog used was Olive Hill flint fire clay calcined to cone 15, and passed through a ten mesh sieve.

The grog as used contained the following sizes of materials:

Size	*percent*
Through 10 mesh	100
10 to 20	38
20 to 40	33
40 to 60	12
60 to 80	4.5
80 to 100	2.5
100 to 200	5
Through 200	5

The clay and grog were mixed in a large ball mill with no pebbles for half an hour. The eighty pound batch was then pugged in a wet pan for fifteen minutes, all specimens of one mixture being made from the same batch. Briquettes ¾ inch by 1 inch by 6 inches were molded in a brass mold and dried at room temperature. The entire set of briquettes (about 1200 in all) were burned at the same time in a coal and coke-fired kiln, the rate of heating being about 50° per hour. The temperatures reached in the first and second burns (cone 7 and cone 10 respectively) were purely accidental. In the remainder of the burns the temperatures reached were cones 13 to 15 with a final "soaking" period of four to six hours. In the first burn the briquettes were placed in saggers; in the remaining burns they were set open fire on the edge and with a small space between the pieces.

This proved to be a severe test of endurance since many of the briquettes became glazed from kiln ash and stuck together. The entire lot had to be reset twice on account of other use for the kiln and thus the briquettes received quite rough handling. Better results could be obtained using a gas kiln, setting the pieces open fire and not unloading the kiln during the investigation.

After each burn, ten briquettes of each mixture were removed from the kiln, and the breaking strength determined. The briquettes were broken in a ten thousand pound Olsen machine, pressure being applied at a uniform rate by means of

a small motor. The force applied was not measured by the balance arm, but by a reducing device with dial reading, devised by Prof. H. F. Moore of the University of Illinois. The accuracy of the dial reading was about 2 percent, and the rate of manipulation was about one briquette per minute.

The modulus of rupture was calculated from the formula:

$$M = \frac{3Fl}{2\,bd^2}$$

Where M = modulus of rupture,
F = breaking load, in pounds,
l = length, in inches, between supports (4½ inches),
b = breadth, in inches,
d = depth, in inches.

After the briquettes had been broken, the half pieces were used for determinations of porosity, etc. They were weighed, saturated with water *in vacuo,* and suspended, and wet weights were determined. The formulæ and designations used are as follows:

P = total pore space in percent of true clay volume;
P_1 = open pore space in percent of true clay volume;
P_2 = closed pore space in percent of true clay volume;
P_3 = open pore space in percent of briquette volume;
D = dry weight; S = suspended weight;
W = wet weight;
S_1 = true specific gravity;
S_2 = apparent specific gravity;
A = absorption, in percent of dry weight.

$$P = P_1 + P_2$$

$$P_1 = 100\,\frac{W\text{-}D}{\frac{D}{S_1}}$$

$$P_2 = 100\left(\frac{S_1}{S_2} - 1\right)$$

$$P_3 = 100\frac{W\text{-}D}{W\text{-}S}$$

$$S_2 = \frac{D}{D\text{-}S}$$

$$A = 100\frac{W\text{-}D}{D}$$

Since true specific gravity is yet to be determined, the only quantities given in this report are P_3, A, S_2, and M. Values for the remaining quantities will be given in the final report.

RESULTS

The average moduli of rupture and average percent deviation from mean are shown in Table II and the results plotted in Fig. 1. The strength of all mixtures increased from burn 1 (cone 7) to burn 2 (cone 10). From burn 2 to burn 3 (cone 15), all except two mixtures decreased in strength and continued, in general, to decrease to burn 10. There is, however, a general lag in the curves from burn 4 to burn 7 which is accompanied by bows in the porosity and apparent specific gravity curves (Figs. 2 and 3).

TABLE I. INCIDENTAL DATA

CLAY	DEFORMATION POINT	WATER IN MIXTURES	DRYING SHRINKAGE OF MIXTURE	BURNING SHRINKAGE OF MIXTURE
	cone	*percent*		
1. Illinois pot clay.....	31	22.5	5.2	3.7
2. Ohio fire clay.....	30	17.1	4.1	1.7
3. Kentucky pot clay.....	31	20.0	4.3	2.2
4. New Jersey fire clay A...	31	19.6	3.6	2.3
5. New Jersey fire clay B...	30	15.7	2.3	0.1
6. St. Louis fire clay.....	30	15.8	4.4	2.0
7. Tennessee ball clay No. 7.	31	22.6	4.4	3.4

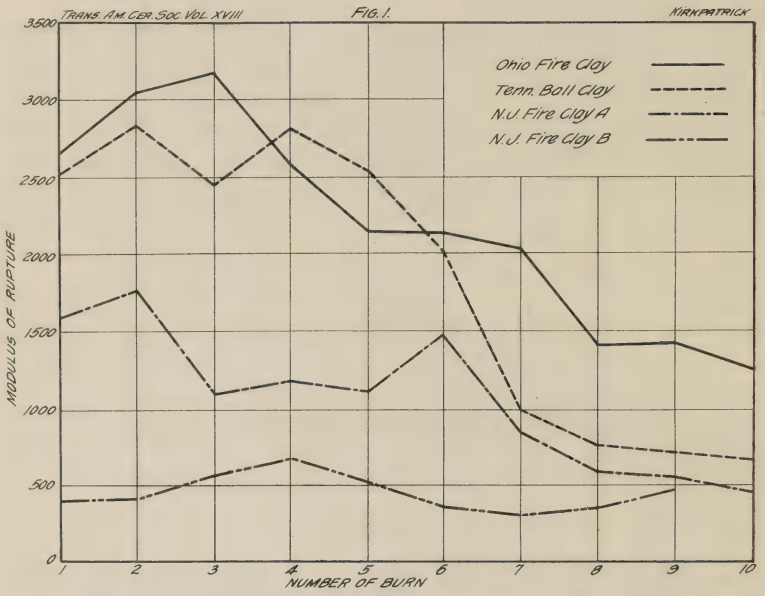

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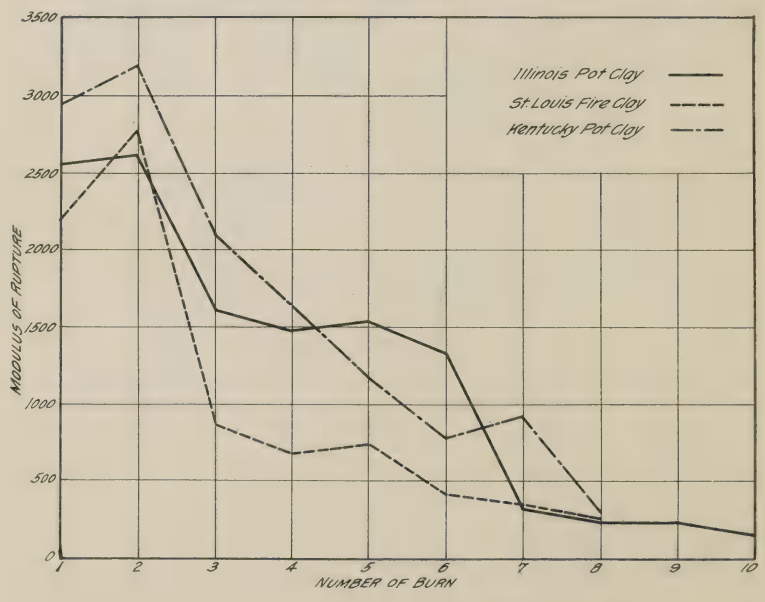
FIG. 1.
KIRKPATRICK
Ohio Fire Clay
Tenn. Ball Clay
N.J. Fire Clay A
N.J. Fire Clay B
MODULUS OF RUPTURE
NUMBER OF BURN
0
500
1000
1500
2000
2500
3000
3500
1
2
3
4
5
6
7
8
9
10


Illinois Pot Clay
St. Louis Fire Clay
Kentucky Pot Clay
MODULUS OF RUPTURE
NUMBER OF BURN
0
500
1000
1500
2000
2500
3000
3500
1
2
3
4
5
6
7
8
9
10

The relation between porosity and modulus of rupture is inconsistent, although it seems to be in general a parallel relation. This is apparently contrary to previous experience. It has been found[3] that there is a linear relation between the absorption and the crushing strength of burned shale, increase of one accompanying decrease of the other. A similar relation might be supposed to exist between porosity and breaking modulus of fire-clay grog mixtures, although it is unsafe to assume such a relation since it has not been established by experimental work. Moreover, the porosities of the fire-clay grog mixtures used were much greater than those of shale bodies, and the changes were small: from burn 1 to burn 4 average decrease, 4 percent; from burn 4 to burn 10 average change, 1 percent. Hence correlation of the two properties is difficult, even with single mixtures.

Mellor[4] found that a clay body on repeated heating decreased in strength, then increased. This phenomenon was observed to a slight degree in the present investigation, each mixture showing a lag or slight increase in strength at some point beyond which the strength again decreased. The lowering in strength from burn 6 to burn 7 was general, corresponding approximately to the length of life of saggers as found by Simcoe.[5]

In Table II it is seen that at burns 3 and 4 the percent deviation from mean of the breaking moduli increased greatly with nearly all mixtures. New Jersey fire clay B showed less deviation from mean and more constant moduli for the entire burning treatment than any other mixture. This was due to its being open grained and tough (non-brittle). On the other hand, the brittle close-grained St. Louis mixtures showed large deviations and really were valueless after burn 5, since half the remaining pieces were cracked after this burn, and only specimens of perfect appearance were chosen for testing.

[5] *Trans. Amer. Cer. Soc., Vol.* XVII, p. 256.
[4] Mellor and Austin, *Trans. Eng. Cer. Soc.*, Vol. VI, p. 129.
[3] Bleininger, *Trans. Amer. Cer. Soc.*, Vol. XII, p. 564.

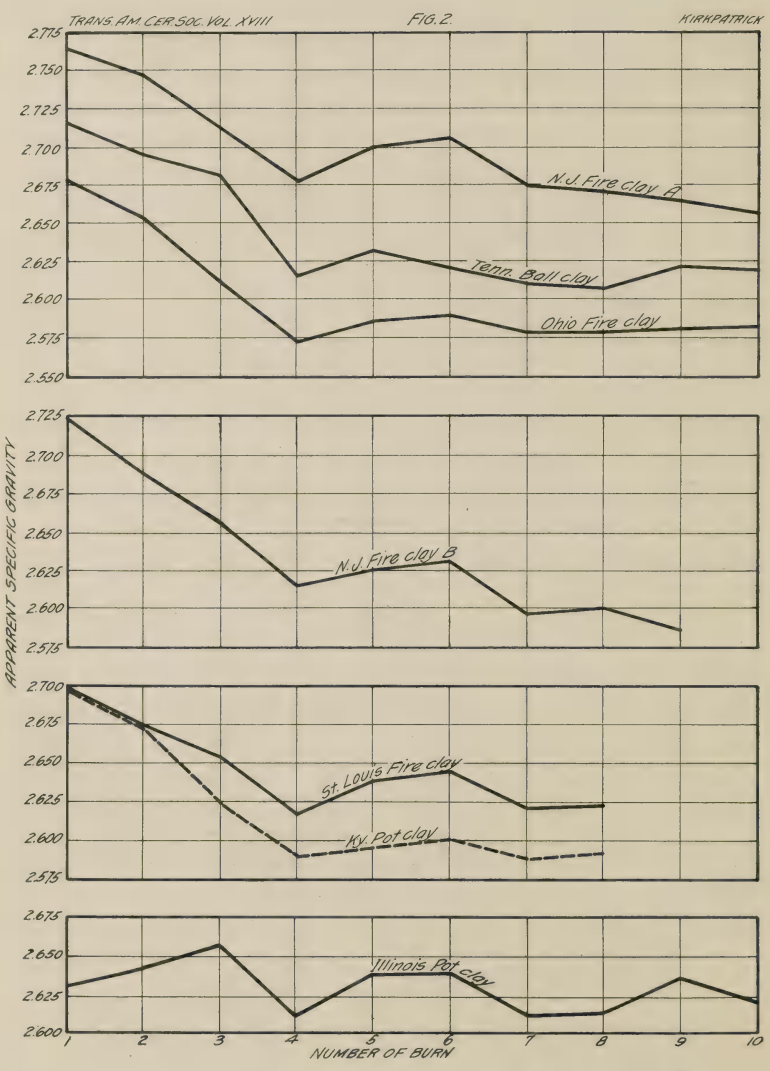

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FIG. 2.
KIRKPATRICK
APPARENT SPECIFIC GRAVITY
N.J. Fire clay A
Tenn. Ball clay
Ohio Fire clay
N.J. Fire clay B
St. Louis Fire clay
Ky. Pot clay
Illinois Pot clay
NUMBER OF BURN

Between these two extremes lie the Ohio fire-clay mixtures, which, while tight burning and somewhat brittle, show a gradual increase in deviation from mean and with little exception higher moduli of rupture throughout the entire ten burns than any other mixture. The remaining mixtures also lie between these two extremes with regard to breaking strength.

TABLE II. MODULI OF RUPTURE AND PERCENT DEVIATION FROM MEAN

NUMBER OF BURN	TEMPERATURE CONE	ILLINOIS POT CLAY	OHIO FIRE CLAY	KENTUCKY POT CLAY	NEW JERSEY FIRE CLAY A	NEW JERSEY FIRE CLAY B	ST. LOUIS FIRE CLAY	TENN. BALL CLAY
1	7	2576	2658	2956	1575	393	2155	2538
		7.7	5.3	3.4	4.0	5.8	12.2	5.8
2	10	2624	3053	3212	1774	386	2829	2859
		9.5	6.7	3.1	6.7	7.8	7.2	6.0
3	15	1596	3188	2113	1062	550	843	2487
		88.0	7.5	38.9	50.3	9.3	41.3	18.0
4	13	1467	2570	1627	1166	627	622	2843
		76.7	26.9	83.5	62.0	11.2	70.2	27.3
5	13	1546	2142	1128	1084	500	727	2531
		72.2	54.1	88.6	65.6	12.6	82.4	31.5
6	13	1327	2132	755	1472	354	377	2020
		85.5	65.2	74.0	28.8	35.8	48.3	46.6
7	14	245	2046	913	812	294	321	981
		71.7	56.8	83.0	65.8	36.6	83.2	63.7
8	14	199	1407	280	584	330	186	733
		79.8	51.3	55.3	30.6	36.2	61.9	57.0
9	14	215	1419	....	544	445	....	689
		47	57.8	....	27.2	26.0	....	44.9
10	14	96	1242	....	453	....	....	640
		56	57.2	....	49.4	....	....	63.5

The values of porosity, absorption and apparent specific gravity are given in Table III and plotted in Figs. 2 and 3. Determinations were made on five speciments of each mixture for each burn. One set of data is given below to afford an idea of the accuracy of the work and of the variation of the values.

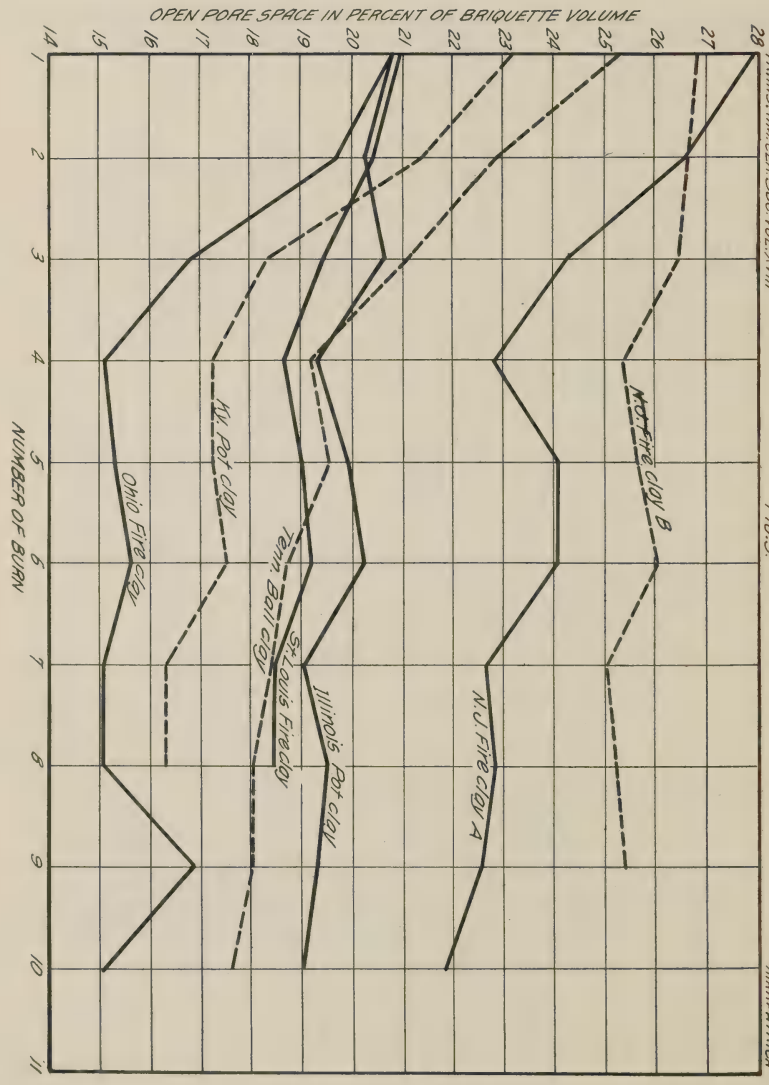
OPEN PORE SPACE IN PERCENT OF BRIQUETTE VOLUME
14
15
16
17
18
19
20
21
22
23
24
25
26
27
28
NUMBER OF BURN
1
2
3
4
5
6
7
8
9
10
11
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FIG. 3
MARKHAM & PATRICK
Ohio Fire clay
Ky. Pot clay
Ann Arbor clay
St. Louis Fire clay
Illinois Pot clay
N.J. Fire clay A
N.Y. Fire clay B

BURN I. ILLINOIS POT CLAY

NO.	D	S	W	POROSITY $\frac{W-D}{W-S}100$	ABSORPTION $\frac{W-D}{D}100$	APPARENT SPECIFIC GRAVITY $\frac{D}{D-S}$
127	89.94	55.60	98.73	20.37	9.77	2.618
159	75.31	46.81	83.41	22.13	10.74	2.644
158	84.87	52.73	93.58	21.32	10.26	2.640
199	77.06	47.69	84.27	19.71	9.35	2.624
198	74.38	46.08	81.71	20.57	9.85	2.628
Average				20.82	9.99	2.631

Neither porosity nor specific gravity changed much after the fourth burn. Explanations of the observed phenomena cannot be attempted until true specific gravity of the specimens has been determined and microscopic examinations have been made.

TABLE III

NUMBER OF BURN	POROSITY P_3 $\frac{W-D}{W-S}.100$	ABSORPTION $\frac{W-D}{D}.100$	APPARENT SPECIFIC GRAVITY $\frac{D}{D-S}$
ILLINOIS POT CLAY			
1	20.83	9.99	2.631
2	20.27	9.89	2.644
3	20.68	9.98	2.656
4	19.32	9.18	2.610
5	19.94	9.45	2.638
6	20.31	9.66	2.641
7	19.10	9.05	2.612
8	19.55	9.17	2.617
9	19.39	9.15	2.626
10	19.10	9.01	2.620
OHIO FIRE CLAY			
1	20.82	9.83	2.672
2	19.76	9.28	2.653
3	16.81	7.62	2.616
4	15.11	6.92	2.571
5	15.32	7.00	2.586
6	15.68	7.18	2.592
7	15.10	6.90	2.577
8	15.10	6.90	2.578
9	16.92	7.65	2.582
10	15.21	6.95	2.583
KENTUCKY POT CLAY			
1	23.18	11.17	2.701
2	21.42	10.24	2.674
3	18.34	8.54	2.625
4	17.24	8.06	2.590
5	17.28	8.05	2.595
6	17.53	8.23	2.600
7	16.35	7.54	2.585
8	16.36	7.53	2.594
9	.....	.....	.....
10	.....	.....	.....
NEW JERSEY FIRE CLAY A			
1	27.95	14.02	2.765
2	26.62	13.07	2.748
3	24.21	11.79	2.711
4	22.80	11.30	2.678
5	24.06	11.81	2.700
6	24.05	11.71	2.705
7	22.64	10.95	2.673
8	22.84	11.06	2.669
9	22.60	10.96	2.664
10	21.89	10.57	2.657

NUMBER OF BURN	POROSITY P_3 $\frac{W-D}{W-S}.100$	ABSORPTION $\frac{W-D}{D}.100$	APPARENT SPECIFIC GRAVITY $\frac{D}{D-S}$
NEW JERSEY FIRE CLAY B			
1	26.89	13.63	2.725
2	26.66	13.52	2.689
3	26.44	13.64	2.655
4	25.32	12.98	2.615
5	25.67	13.16	2.626
6	26.02	13.37	2.631
7	25.05	12.88	2.595
8	25.29	13.00	2.600
9	25.49	13.21	2.589
10	.....	.....	.....
ST. LOUIS FIRE CLAY			
1	20.94	9.86	2.700
2	20.42	9.59	2.673
3	19.42	9.08	2.654
4	18.70	8.81	2.618
5	19.02	8.90	2.642
6	19.22	9.06	2.645
7	18.50	8.66	2.621
8	18.50	8.66	2.623
9	.....	.....	.....
10	.....	.....	.....
TENN. BALL CLAY			
1	25.27	12.21	2.718
2	22.85	10.99	2.694
3	21.11	9.98	2.682
4	19.14	9.05	2.614
5	19.54	9.23	2.633
6	18.73	8.79	2.621
7	18.42	8.66	2.613
8	18.02	8.42	2.609
9	18.07	8.42	2.622
10	17.56	8.13	2.620
TEMPERATURES CONE			
1	7	.....	.....
2	10	.....	.....
3	15	.....	.....
4	13	.....	.....
5	13	.....	.....
6	13	.....	.....
7	14	.....	.....
8	14	.....	.....
9	14	.....	.....
10	14	.....	.....

VARIATION IN LINEAR SHRINKAGE OF CLAY TEST PIECES

BY JAMES BAILEY

The shrinkage of a specimen of clay or batch may be determined by either of two well known methods.

The volume method, which consists of measuring the wet and dry volumes of the clay and computing the percent of the volume shrinkage, has been used as the standard method by many scientific investigators, but because of its tediousness it has not been much used by manufacturers. It is customary to simply shape the clay into a bar or slab, mark it with fine reference lines, measure the original distance between these lines and again measure it after the specimen has reached its final size. The difference expressed in percent is called the linear shrinkage. Variations in the results obtained by the latter method are those referred to in this paper.

Preliminary experiments showed that the most important causes of variations in the linear shrinkage of any given batch were due to, first, the method of making the test piece, and, second, to the change in the moisture content while working the batch and making the specimens.

The distribution of the grog was found to be so uniform that no variations could be traced to this cause, as was also the sizing of the grog particles and moisture distribution. Variations due to the drying out of the batch while handling were much too small to cause the wide variations in the results. For reference, however, the moisture content of each specimen should be determined. The chief cause was found to be the method of shaping the test pieces, and a series of experiments was therefore conducted to show the effect upon the linear shrinkage of different methods of making the specimens.

Four methods were selected as follows:

First method: "Rolled." A large piece of the well mixed batch was cut into pieces of suitable size. The pieces were then rolled into cylinders between the hands and a flat surface. Each

TRANS. AM. CER. SOC. VOL. XVIII FIG. 1. BAILEY

Mark	Wet Length	Moisture Percent of Dry Weight	Drying Shrinkage Percent of Dry Length	Burning Shrinkage Percent of Dry Length	How Made
1	7"	19.2	6.55	1.2	Rolled
2	6"	18.8	5.83	1.04	
3	7"	18.73	5.90	1.31	
4	7"	18.75	6.22	1.43	
5	7"	18.9	6.38	1.6	
Av.			6.17	1.316	
6	5"	18.7	4.82	1.63	Pounded
7	6"	19.4	4.90	1.78	
8	6"	19.4	5.07	1.58	
9	10"	19.2	4.38	1.48	
10	8"	19.3	4.98	1.54	
Av.			4.83	1.60	
11	6"	19.2	7.15	1.62	Stretched Lengthwise
12	5"	19.3	8.70	1.62	
13	7"	19.1	7.70	1.64	
14	6"	19.0	7.15	1.62	
15	7"	19.2	7.36	1.80	
Av.			7.61	1.66	
16	5"	18.1	2.04	.16	Compressed Lengthwise
17	5"	18.2	3.52	.163	
18	6"	18.3	3.09	.202	
19	6"	18.3	2.92	.337	
20	5"	18.3	3.10	.000	
Av.			2.93	.123	

cylinder was then weighed, placed upon a flat surface covered with tissue paper and immediately marked with two fine lines about seven inches apart. In handling the cylinders, great care was taken to prevent any stretching or bending of the soft clay.

Second method: "Pounded." The pieces were first made into cylinders as above, but were lightly pounded with the heel of the hand until they were decidedly flattened. They were then weighed, placed upon the tissue paper and marked as before.

Third method: "Stretched Lengthwise." The rolled cylinders were stretched lengthwise about two inches just before being placed upon the tissue paper and marked.

Fourth method: "Compressed." The cylinders were compressed lengthwise by slight pressure on the ends until the surface showed signs of wrinkling. They were then weighed, placed upon the tissue paper and marked.

All specimens were allowed to dry in the open air until bone dry. They were then placed in an oven and dried to constant weight at 110° C., after which they were weighed, and their dry measurement taken.

The specimens were burned together in a small experimental kiln to cone 12 in approximately eight hours.

Results: The results are given in Fig. 1, and the average graphically shown in Fig. 2, together with the mean value determined by computation from the volume shrinkage.

Inspection of the table shows that the variations caused by the different moisture contents are insignificant compared with other factors. It will also be seen that the data for each of the four groups is entirely distinct, *i. e.* the higest shrinkage in one group is smaller than the lowest in the group having the next highest average and *vice versa.*

Figure 2 is self explanatory. The average measured drying linear shrinkage of the rolled specimens is 21 percent higher than the mean shrinkage determined by the volume method. The pounded specimens gave slightly lower values than the mean. Stretching the cylinders caused the measured shrinkage to exceed the mean value by 50 percent and compressing them lengthwise caused the measured shrinkage to fall to 42 percent of the mean value. The difference between the measured shrinkage of the specimens stretched lengthwise and that of the specimens compressed lengthwise is nearly equal to the total mean drying shrinkage.

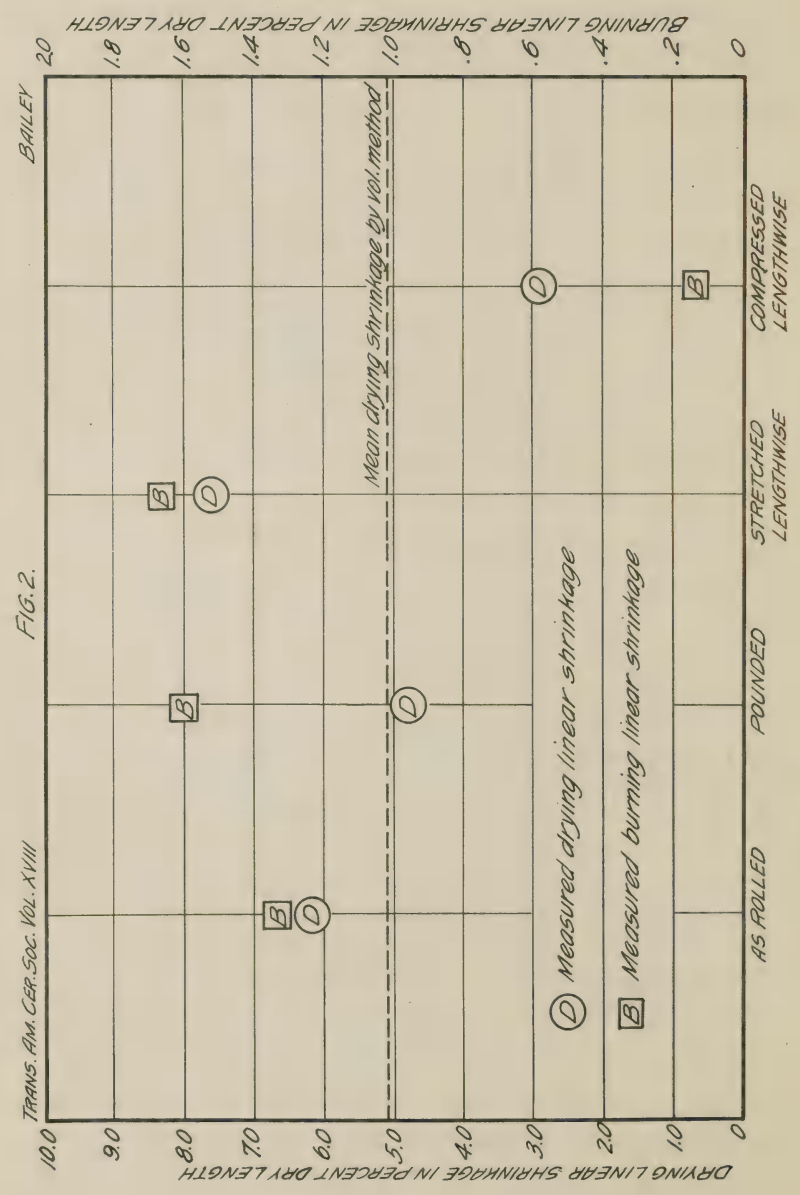
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FIG. 2.
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DRYING LINEAR SHRINKAGE IN PERCENT DRY LENGTH
BURNING LINEAR SHRINKAGE IN PERCENT DRY LENGTH
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
2.0
1.8
1.6
1.4
1.2
1.0
.8
.6
.4
.2
0
Mean drying shrinkage by vol. method
Measured drying linear shrinkage
Measured burning linear shrinkage
AS ROLLED
POUNDED
STRETCHED LENGTHWISE
COMPRESSED LENGTHWISE

It may also be noted that, except in the case of the pounded specimens, the burning shrinkage is increased when the drying shrinkage is increased, and *vice versa*.

Explanation of the Phenomena: A clay or clay batch is made up of hard particles separated by thin layers of water and colloidal substance. The colloidal substance when wet is a jelly-like mass capable of flowing under pressure while the hard particles remain unchanged. Pressure in any direction but not in all directions at the same time squeezes the water and colloidal substance from between the hard particles along the line of pressure until the hard particles come into contact. But very slight pressure causes this to take place. Further pressure displaces the hard particles and in the case of batches containing large grog particles, a grating sound may be heard.

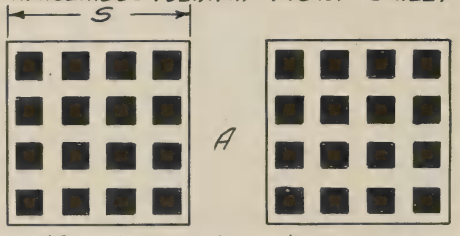
It is evident that the colloidal films are thinner along the line of pressure and hence are thicker at right angles to the line of the applied force, and since it is the aggregate shrinkage of these films that is measured, the measured shrinkage is less along the line of the applied force than it is at right angles to it.

This rearrangement of the colloidal films is pictorially represented in Figure 3.

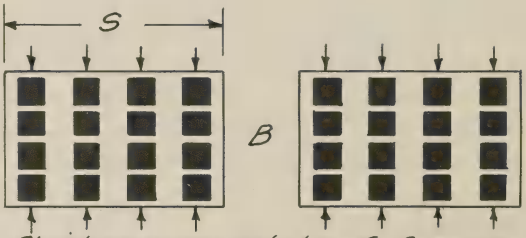
The upper left hand cubes show the uniformly mixed batch, the black squares representing the hard particles and the white spaces, the plastic substance. The shrinkage is measured along *S* and is the same in all other directions. If we call the hard particles (black) grog and the plastic substance between (white spaces) clay, the ratio of clay to grog in any direction is .66.

If a pressure is exerted upon the top and bottom of this cube, the cube is contracted vertically and expanded horizontally into the shape shown in the upper right hand corner. Here the arrows show the direction of the applied force and the shrinkage is measured along *S* as before. The vertical contraction has caused the white spaces along the line of pressure to diminish and those at right angles to it to become thicker. The ratio of clay to grog along *S* has been increased from .66 to 1.0. If *S* were measured vertically it would be reduced considerably below .66.

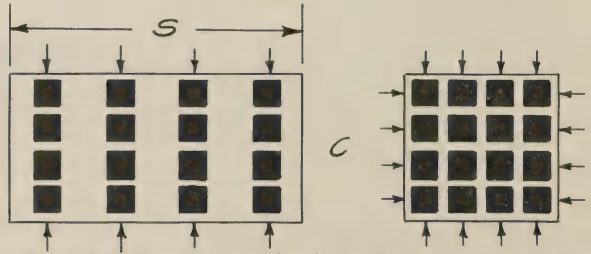
TRANS. AM. CER. SOC. VOL. XVIII FIG. 3. BAILEY



Uniformly mixed. Ratio Clay to Grog along S = .66



Shrinkage measured along S. Compressed in one other direction. Ratio Clay to Grog along S = 1.0



Shrinkage measured along S. Compressed in both other directions. Ratio Clay to Grog along S = 1.66

Another force acting upon this flattened cube as shown in the lower figure causes a further flowage of the plastic substance again increasing the white spaces along *S*, and raising the ratio of clay to grog to 1.66.

Figure 3-A represents the mean value found by computation from the volume shrinkage. Rolling, which is a series of compressions along any diameter, tends to bring the specimen into the condition of Fig. 3-C, as also does stretching lengthwise. Compressing lengthwise brings the specimen into the condition illustrated in Fig. 3-B, but the shrinkage would be measured vertically instead of horizontally as shown.

Conclusion: Although the four methods of making the specimens used in these experiments were intended to develop the maximum variations, the forces required to start the displacement of the plastic substance is so small that it is impossible to handle the specimens without causing a change in the measured linear shrinkage. The shrinkage is usually different in different parts of the same specimen. As a means of determining the properties of a clay for record or comparison with other clays, the direct measurement method is very unsatisfactory. It is the belief of the author that the more accurate results obtained by the volume method will more than repay the experimentor for his added trouble.

AN APPARATUS FOR STUDYING THE DRYING BEHAVIOR OF CLAYS

BY M. GROVER BABCOCK

It is well known that the greater percentage of the aeolin (loess) clays found in the middle west are commercially worthless, due to their inability to withstand dryer conditions. In view of these conditions, the Engineering Experiment Station of the state of Iowa furnished the funds for a study of the drying of clays, more especially those of its own state. An apparatus for studying the drying of clays was designed and built to enable the writer to observe carefully the changes which took place in the process. As this apparatus may be applied to drying nearly all clays, the author is presenting a diagramatic sketch (not drawn to scale) of the apparatus, and the following detailed description.

In attempting to carry out a series of experiments on the drying behavior of clays, there are several important conditions which, due to their nature, must be under direct control of the investigator, *i. e.,* temperature, humidity, and velocity of the air. This apparatus handles the aforesaid conditions in a fairly satisfactory manner.

In general, the apparatus consists of an expansion tank, air regulator, gas meter, pre-heater and drying chamber, all being connected in a series by suitable piping. Provision is made in several places for the insertion of thermometers and other recording instruments.

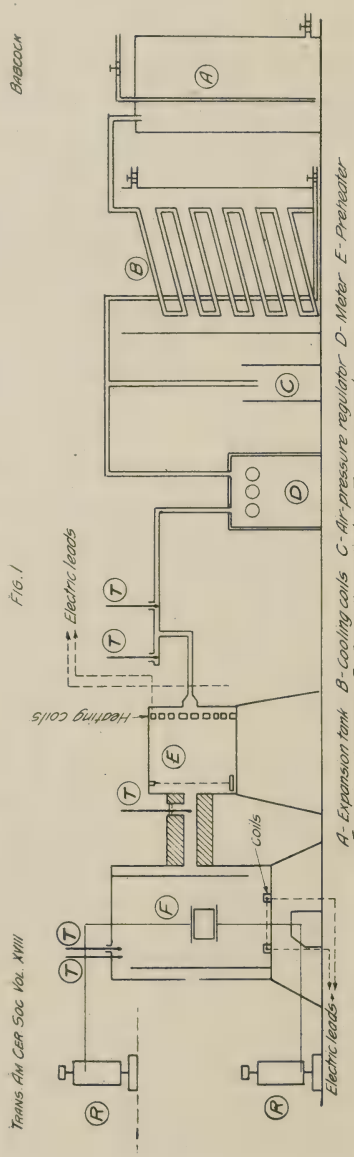
The air leaving the compressed air line of the laboratory enters the expansion tank and cools slightly, due to the release of pressure. If any undue moisture is present, it condenses and may be drawn off at this time by a drip pipe from the tank. The air passes through one-half inch pipe into a cooling coil surrounded by running water, to insure constant temperature and humidity of the air. If desired, a freezing solution may be used instead of the tap water, and a minimum humidity produced.

Any excess water condenses in the coil and is drawn out through the bottom by means of a drip pipe.

The flow of the air is regulated by means of a pipe extending from the line connecting the meter and coils into a bottle of coal oil, which is immersed to a definite depth. A slight excess of air is always kept present and escapes through the oil in bubbles. The higher the pressure, the greater the volume of air escaping. The air that does not escape through the regulator flows through an ordinary gas meter, calibrated to cubic feet and multiples thereof. Here both the volume of air and its rate of flow are measured. The volume may be read from the recording dial, and the rate of flow can be calculated by timing the movement of the meter hands through average time intervals.

There are located, in the piping connections of the meter and on the heating chamber, suitable openings for the insertion of wet and dry bulb thermometers, thereby enabling the operator to measure the temperature and humidity of the air used in drying. Traveling through the piping from the meter, the air comes to the air heater or pre-heater, and here it is raised to the temperature that is desired for the drying. The heater itself consists of a galvanized iron box, heavily lagged with asbestos. In one end is a heating unit made of nichrome resistance wire and connected in circuit with a 220 volt electric main. This furnishes the heat required to raise the air to any temperature up to 330° F. The temperature is regulated by means of a contact breaker made of an alloy of two metals which bends away from the fixed point according to the degree of heat affecting the linear expansion. This thermostat works very well. Temperatures may be held constant to 1° for a period of several hours.

In one end of the heater there is provided a small pan 2 in. by 10 in. by ¾ in., and in front of the exit opening, a piece of burlap is suspended from the top terminating in the pan of the heater. When hot air of high moisture content is wanted, steam in regulated amounts is passed into the pre-heater above the pan



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and in part condenses in it. The moisture passes up by capillary action through the burlap which acts as a wick. As the hot air from the back end of the heater passes through the open structure of the burlap curtain, it takes up the water. From the heater the air passes through a 2½ in. duct, which is heavily lagged with steam-pipe asbestos to prevent radiation into the dryer proper. Here it is forced to flow down around a partition wall to the bottom of the dryer and is then allowed to fill the open space. Thus any air currents are broken up. There is a similar partition wall on the opposite side with an opening at the top, and the exit air passes through this space accomplishing the same purpose in regard to air currents.

The inside dimensions of the dryer are 10 in. by 10 in. by 14 in., it is constructed of ½ inch asbestos board. The recording devices are similar to those reported by Professor Bleininger in his paper on "An Oven with Recording Attachments," Vol. XVI, *Trans. Amer. Cer. Society.* In fact, we have used his ideas for the inner workings of the dryer with the following exception. There is a resistance coil resting on the floor of this dryer which serves as an auxiliary heater, its principal advantage being an aid to the control of the temperature. In attempting to work without this auxiliary heater, it was found that there was a variable drop in the temperature of the air between the pre-heater and the dryer. It is used also to study the effect on the cracking of clays, of slow and rapid heating up, without air currents. The writer would refer those who wish a more detailed description as to the construction of the inner parts of the dryer, to Professor Bleininger's paper. The loss of weight and shrinkage of the two samples are automatically recorded at the same time. The resulting curves of loss in weight and shrinkage are greatly magnified due to the mechanical construction.

In operating the dryer, the clays used are weighed out dry, and enough water is added to give them a good working plasticity, usually from 20 to 25 percent. The clay is shaped in a cylindrical die 2½ in. by 2½ in. The samples weigh about 1½

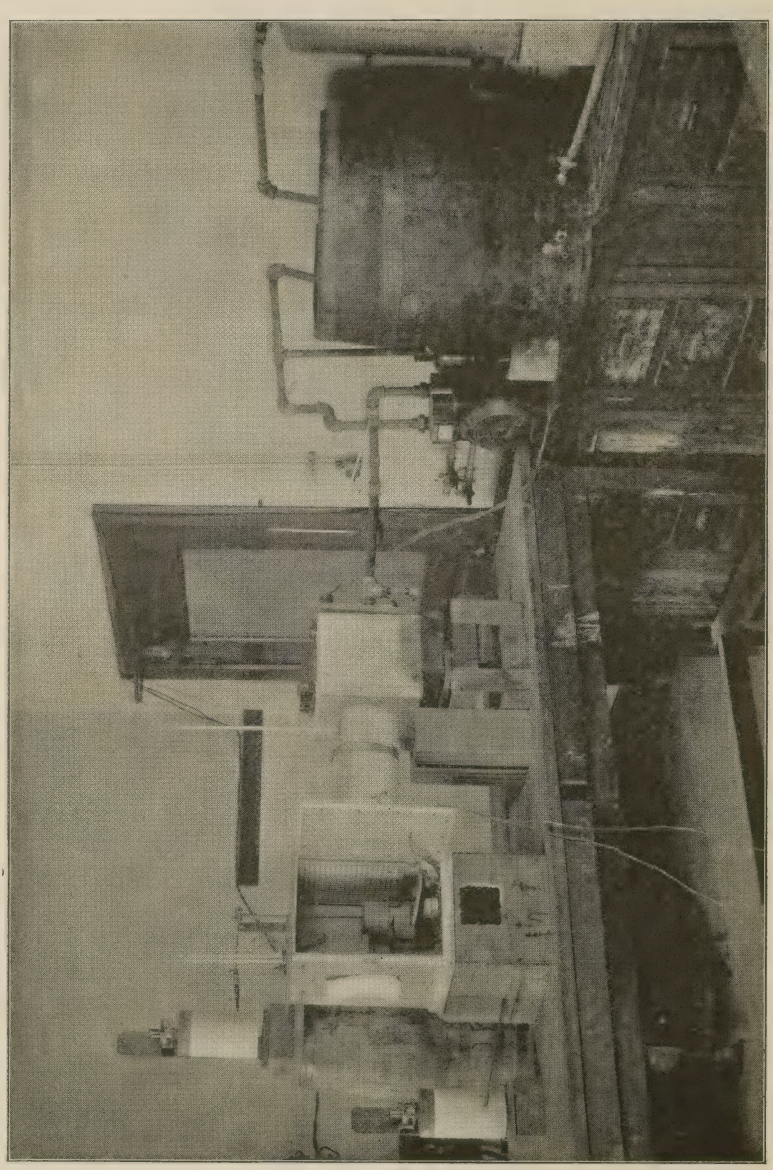

FIG. 2.

pounds wet weight; they are submitted to a pressure of 400 pounds per square inch by means of a hydraulic press with an automatic release to insure the same conditions of structure in every sample.

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HEAT TRANSMISSION OF ENAMEL

BY EMERSON P. POSTE.

An attempt to locate data on the heat transmission of enameled steel as compared with that of copper and plain steel does not result in findings of great value, and the actual conditions, under which recorded tests have been made, vary so that it is difficult to correlate results. It is generally accepted that the nature of the surface presented to the heating medium has much to do with the rate of transmission. Nevertheless, experimental work has been done entirely ignoring this difference and making no effort to have a uniform nature of surface.

Aside from a purely theoretical point of view, it is commercially necessary for the manufacturer of enameled apparatus to know the effect of the enamel on the time required to do a given amount of heating, especially when the apparatus under consideration is to replace similar apparatus of copper or plain steel.

We have clearly two problems. First, what is the effect of the enamel under uniform conditions of surface exposed to the heating medium; secondly, what bearing has the difference of surface condition on the practical results to be obtained. Only the first phase of the subject is to receive attention here.

Two general conditions come to mind as we attack this problem. The first has to do with the actual effect of varying thickness of enamel coating and the second with the heat transmission of enameled steel as compared with that of other materials which it may be called upon to replace. The observation recorded in this paper throw light on these two phases of the problem.

To cover the first point it seemed reasonable to make use of one set of heating conditions throughout, but from a practical point of view it seemed necessary to include three sets of conditions in studying the second point. We may have as a

heating medium a hot liquid transmitting heat to a liquid not boiling; secondly, by means of steam we may heat a liquid which is not boiling and finally we may have a boiling liquid heated by steam. There are various other possibilities, but the above seem sufficient to include in a general investigation.

The experimental work included series of tests under the general conditions outlined above. The trials with which these tests were made were small cylindrical dishes 3 in. deep by 5 in. in diameter with a half-inch flange at the top and uniform thickness of metal. They were filled with a known weight of water, and the water evaporated by exposure of the dishes to the various heating media. The time to completely evaporate the given amount of water was observed.

The effect of varying the thickness of the enameled coating was noted by placing in a boiling water-bath the following dishes, each of which contained 500 cc. of water:

A — Plain steel.
B — One coat of enamel.
C — Two coats.
D — Three coats.
E — Four coats.
F — Five coats.
G — Six coats.

The outside as well as the inside of each dish had been sand-blasted to assure a uniform quality of surface exposed to the heating medium. The time required to evaporate the water in each dish was noted. The results follow:

A — 128 minutes.
B — 151 minutes.
C — 157 minutes.
D — 168 minutes.
E — 183 minutes.
F — 186 minutes.
G — 199 minutes.

A comparison of these figures is best shown by Figure 1.

The comparison of copper, unenameled steel, and enameled steel was carried out under the three sets of conditions outlined above. For the first comparison the following dishes were placed in a boiling water-bath and the time to evaporate 500 cc. was noted.

A — Copper.
B — Plain steel.
C — Enameled steel, 4 coats.

As before, all had been sand-blasted outside and inside. The time required to evaporate the water was as follows:

A — 140 minutes.
B — 153 minutes.
C — 179 minutes.

Curve 1 on Figure 2 gives a comparison of these results.

The second comparison was made using the same three dishes mounted in an apparatus making possible the use of steam at atmospheric pressure as the heating medium.

In each dish was placed 500 cc. of water, and the time required to evaporate to dryness was noted. The results are as follows:

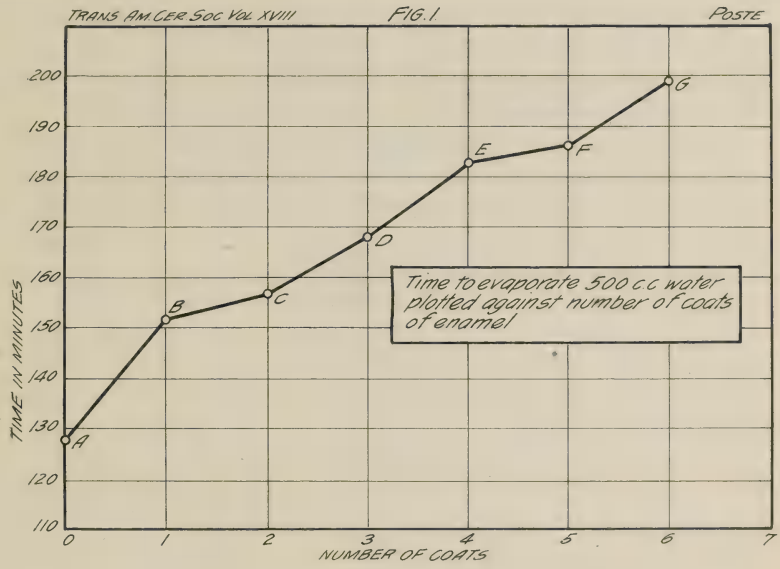
A — 106 minutes.
B — 117 minutes.
C — 164 minutes.

These results are shown on Curve 2, Figure 2.

The third comparison was made in a manner similar to the second with the steam pressure at ten pounds. In this case the water boiled quite vigorously in all the dishes. The results are as follows:

A — 16½ minutes.
B — 19 minutes.
C — 73 minutes.

These values are plotted on Curve 3.


TRANS AM CER SOC VOL XVIII
FIG. 1
POSTE
TIME IN MINUTES
200
190
180
170
160
150
140
130
120
110
0
1
2
3
4
5
6
7
NUMBER OF COATS
A
B
C
D
E
F
G
Time to evaporate 500 c c water plotted against number of coats of enamel

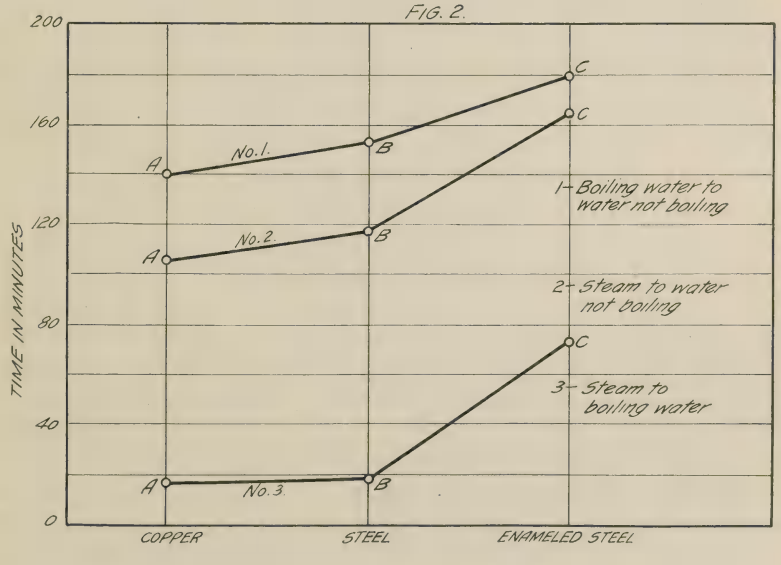


FIG. 2.
TIME IN MINUTES
200
160
120
80
40
0
No. 1.
No. 2.
No. 3.
A
B
C
1- Boiling water to water not boiling
2- Steam to water not boiling
3- Steam to boiling water
COPPER
STEEL
ENAMELED STEEL

The above data leads to the general conclusion that an enamel coating on steel produces an appreciable retarding effect on heat transmissions, which is but slight with hot water as the heating medium but increases as the heating medium is steam under rising pressure.

Experience with commercial apparatus leads to the conclusion that on larger units the difference between copper and enameled steel is not so great as would be concluded from the above tests on an experimental scale. Data on commercial equipment is being obtained and will be included in a later paper on the above subject.

A FUSION STUDY OF THE MINERAL SYSTEMS FELDSPAR CALCITE AND FELDSPAR MAGNESITE[1]

BY F. A. KIRKPATRICK, URBANA, ILL.

INTRODUCTION

Fusion Studies in General. Fusion studies have proven to be valuable aids in controlling the properties of ceramic products. There are many different kinds of fusion studies. Among those of most value to the ceramist are the following:

1. Determination of melting-points of mixtures of minerals.
2. Study of compounds formed and of equilibrium relations in mineral systems.
3. Determination of deformation points of minerals and of mixtures of minerals.
4. Study of viscosity range of substances and mixtures.

Scope of Work. The present investigation is a study of the behavior of the following mineral systems at high temperatures as indicated by the manner, the rate, and the temperature of softening or deformation: —

Orthoclase-Calcite,
Orthoclase-Magnesite,
Albite-Calcite,
Albite-Magnesite.

The literature on fusion studies and related phenomena is too great in extent to allow of a review at this time. References will be given as required from the partial list at the end of this report.

Deformation Point. The deformation point of a body is that temperature at which it deforms on heating. For this de-

[1] Abstract of thesis done in partial fulfillment of the requirements for the degree of Master of Science in Ceramic Engineering, University of Illinois, 1916.

termination, the method most commonly used and the one employed in this investigation is as follows: a triangular upright cone of the substance is heated at a definite and uniform rate until it bends so that the top touches the base support, or failing to bend, it fuses to a ball. The temperature at the instant of contact or of balling-up is called the deformation point and is measured by means of a thermocouple pyrometer, optical pyrometer or standard cones. It is understood that in the case of balling-up, repeated trials have shown that the cone possesses such high viscosity and surface tension that it cannot be made to deform normally.

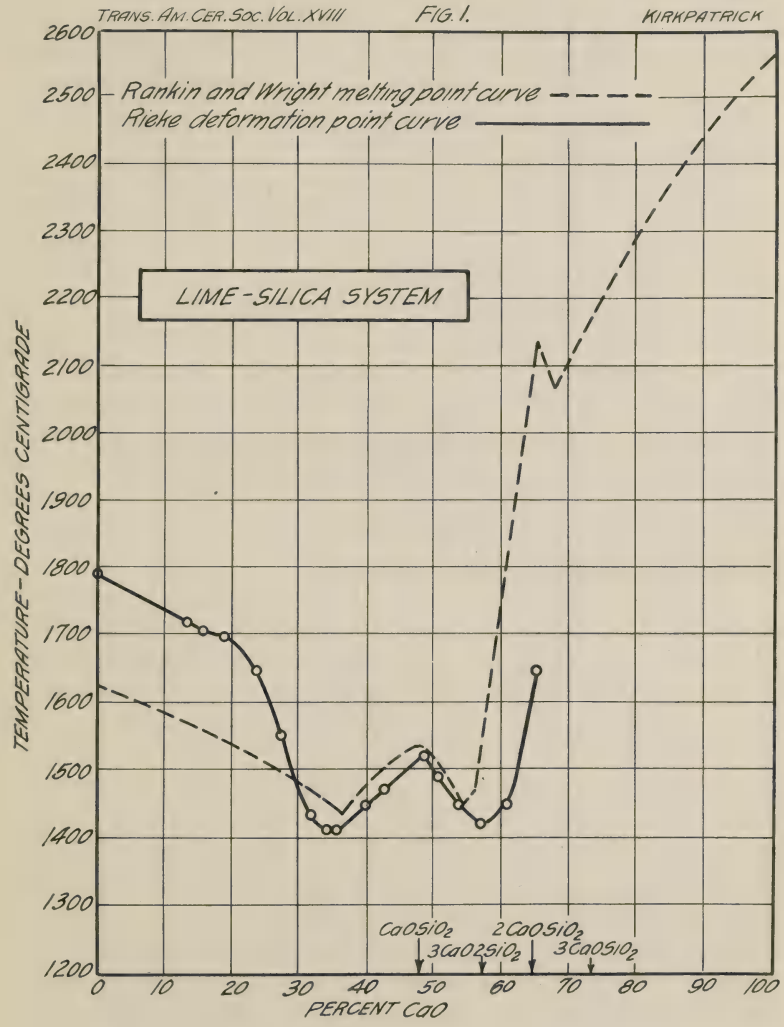
The deformation point is sometimes called the softening point, and in some instances it is necessary to distinguish between the two, as in the case of glass. The softening point curve of glass bears no general relation to the melting point curve or to the deformation point curve of the corresponding raw materials. It generally lies below them.[2]

Melting Point and Melting Point Diagrams. The melting point of a substance is that temperature at which the crystalline phase is in equilibrium with the liquid phase at the existing pressure. The melting point of the substance may be lowered by the addition of another substance. If the two substances are mixed in various proportions and the mixtures subjected to heat changes, the phenomena observed may be represented by means of diagrams.

Melting point and equilibrium diagrams are discussed in detail in so many texts that the reader is referred to these for explanations of such diagrams.

Relation Between Melting Point and Deformation Point. Considering all substances, the melting point is much the same as the deformation point and the substances become fluid at this temperature. Most silicates, however, are quite viscous within a range of temperature above and below their melting points, hence their deformation points may be either above or below. This is shown in Figures 1, 2 and 3. Fig. 1 is the same as that of Staley[3] except that the 1915 curve of Rankin and

[2] Staley, *Ref.* 21, p. 674.
[3] *Ref.* 21


TRANS. AM. CER. SOC. VOL. XVIII
FIG. 1.
KIRKPATRICK
Rankin and Wright melting point curve
Rieke deformation point curve
LIME-SILICA SYSTEM
TEMPERATURE-DEGREES CENTIGRADE
2600
2500
2400
2300
2200
2100
2000
1900
1800
1700
1600
1500
1400
1300
1200
0
10
20
30
40
50
60
70
80
90
100
PERCENT CaO
CaOSiO2
3CaO2SiO2
2CaOSiO2
3CaOSiO2

Wright[4] for CaO-SiO_2 is used, and Rieke's deformation points[5] have been recalculated according to the 1910 Reichsanstalt cone calibration which, from cone 14 up, corresponds to the rate of heating used by Rieke (4° to 5° per min.) It is noted that SiO_2 and mixtures up to 30 percent deform above the melting point, mixtures from 30 percent to 58 percent deform at from 10° to 50° below, while the compound $2CaOSiO_2$ at 65 percent CaO deforms 480° below its melting point.

In Fig. 2 are shown melting point and deformation point curves for the system CaO-Al_2O_3. Two facts are to be noted:

1. High lime mixtures deform much below their melting point, as in the system CaO-SiO_2.

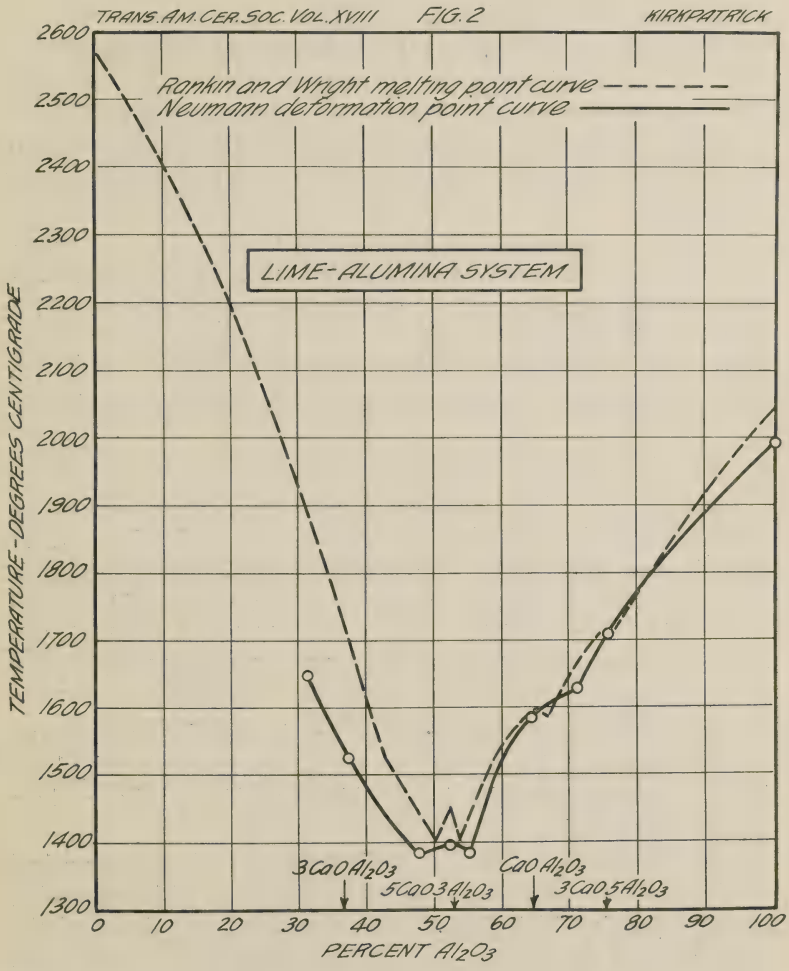
2. Deformation points in the mixtures from 45 to 55 percent Al_2O_3 are below the melting points of the melting point eutectics, resembling the part of the CaO-SiO_2 system (Fig. 1) from 35 to 55 percent CaO. The reason for this is probably that the heat of formation of compounds formed in these mixtures causes the cones to deform, while the Seger cones (by means of which the temperatures are measured) are subject to no such heat effect, and the test cones go down with lower Seger cones than they would otherwise.[6]

In Fig. 3, are shown melting point and deformation point curves for the system SiO_2-Al_2O_3 in which lie the compositions of Seger cones 28 to 42. It is noted that mixtures up to 80 percent of Al_2O_3 deform above the melting point, the remainder below the melting point. It is observed that deformation points of Seger cones made of kaolin and quartz or alumina, are almost identical with those of cones made from the oxides Al_2O_3 and SiO_2. This indicates that the nature of the resultant fusion is the same in both cases. Hence, it may be said that in general the deformation point curve affords an inaccurate means of determining the course of a melting point curve.

Comparison of Value of Melting Point and Deformation Point. The value of knowledge of the deformation point of a ceramic material lies in the fact that it indicates the temperature at which the body softens, regardless of its melting point.

[4] *Ref.* 46.
[5] *Ref.* 58.
[6] *Ref.* 47, p. 1092.

The melting points of many substances cannot be determined on account of high viscosity, supercooling, and slow rate of crystallization, while those of other substances and mixtures



have been determined accurately. Such determinations, in connection with studies of the equilibrium relations have proven valuable, in both practical and scientific work. One need only

cite the work of the Geophysical Laboratory of the Carnegie Institute and of the U. S. Bureau of Standards, on various systems, especially their application of the equilibrium diagram of the CaO-Al_2O_3-SiO_2 system to problems involving the behavior of Portland cement clinker.

Eutectics and Maximum Points. The "melting-point" eutectic has already been defined as that mixture of compounds having the lowest melting point. The "deformation-point" eutectic is any mixture of two substances having a lower deformation-point than neighboring mixtures. It is not capable of exact definition.

Wilson[7] has suggested for compositions at and near maximum points on deformation curves the name "deformation compound", in contradistinction to "deformation-point eutectic", a term brought forward by Staley.[8] Since in many cases the maximum point corresponds to no compound formation, the term "deformation-point maximum" would seem more appropriate.

FACTORS AFFECTING DEFORMATION POINTS OF CONES

Viscosity. The viscosity of many silicates causes them to become fluid slowly instead of suddenly as in the case of most known substances. If some silicates did not possess this property, the production of many ceramic wares would be very difficult if not impossible, and there would be no need of deformation-point determinations since this point would correspond to the melting point.

In general, viscosity decreases with rise in temperature. Cones of mixtures having a slow rate of change of viscosity have long softening intervals those with a rapid rate have short softening intervals.

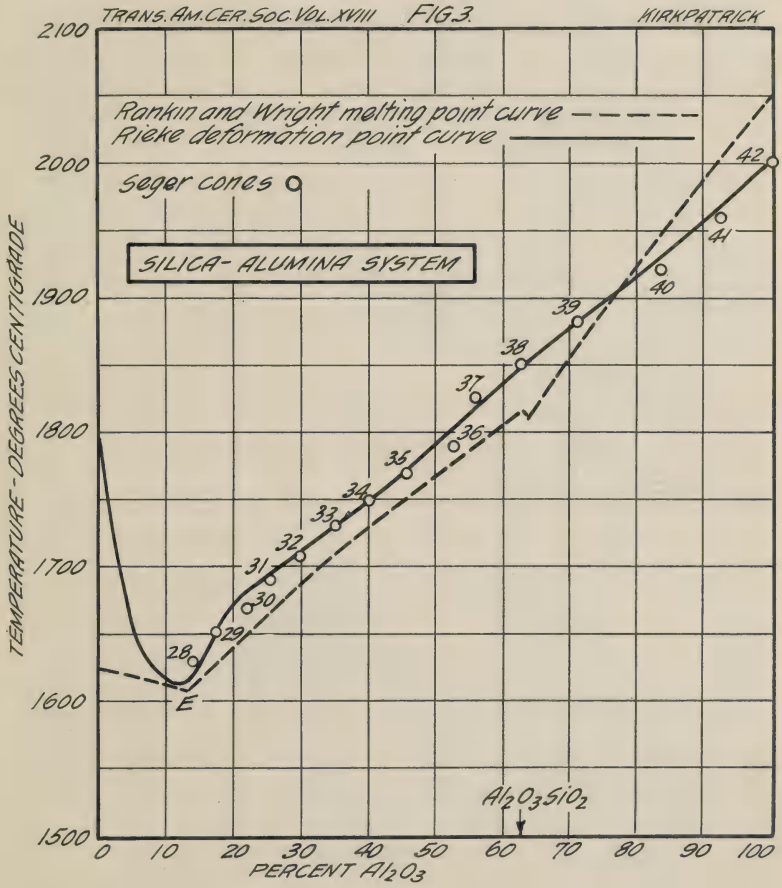
Cones of mixtures having high surface tensions form a ball on the end before bending or sometimes enlarge without bending giving somewhat high deformation temperatures. Some of the cones used in the present investigation behaved in this manner.

Melting Point Eutectic. The effect of the melting point eutectic depends upon the following factors: —

[7] *Ref. 31*, p. 226.
[8] *Ref. 21.*

1. Amount of melting point eutectic present and its viscosity.

2. Amount of excess component present in the liquid state and its viscosity.



3. Total amount of liquid present and its viscosity.

4. Amount of liquid required to cause deformation.

5. The time effect.

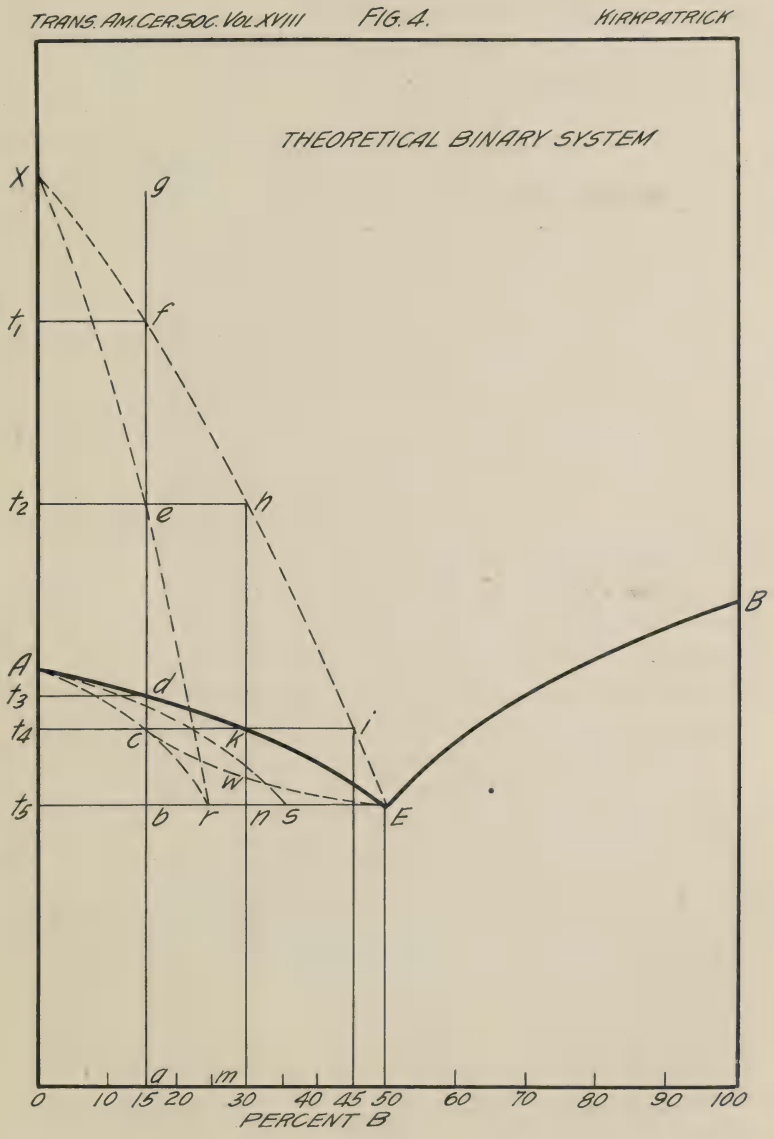
In Fig. 4 it can be shown by means of the lever relation that if the mixtures deformed when 50 percent melted, the

deformation temperatures would lie on the line **AcrE**; if 75 percent is melted, on the line **AsE**; if 100 percent is melted, on the line **AdE**. This takes into consideration only the unmodified effect of the melting point eutectic. The time effect, however, changes the course of these lines, since the temperature during the period of deformation does not remain constant as would be indicated by that part of the deformation point curve from **r** to **E**. Hence the curve would be closer to **AcwE**. Still another factor must be considered, i. e., the viscosity of the excess component **A**. If this is less than the viscosity of the eutectic mixture, the deformation curve will remain far below the melting point curve as in the CaO-SiO_2 system, (Fig. 3) from 60 percent to 65 percent CaO. If the viscosity of the excess component is greater than that of the eutectic, the deformation point curve will remain above the melting point curve as in the CaO-SiO_2 system from 0 to 30 percent CaO.

If the melting point curve were **EX** instead of **EA**, the above effects would be accentuated. Under constant viscosity, the steeper the melting point curve the farther apart are this curve and the deformation point curve, as may be seen by comparison of the "50 percent" curves **AcrE** and **XerE** with the corresponding melting point curves.

The effect of melting point eutectic was well shown by Sosman[9] in the system SiO_2-Al_2O_3 (See Fig. 3). Sosman caused cones 28, 29, 30 and 31 to deform by five hours heating at about 1616° C., a temperature just above eutectic *E*. Cone 33 was one-fourth down, and cone 34 had started to bend. Microscopic examination of cone 34 showed it to contain about 50 percent of crystallized sillimanite and 50 percent of glass. Using the lever relation in connection with Fig. 3, it is found that at 1616°C., cone 34 should contain 46 percent of eutectic E. This shows that all the eutectic had melted but was not liquid enough to bring down the cone in five hours. Examination of the remaining cones would undoubtedly have shown that the eutectic (larger percentages of the mass, of course) had all melted and that very little of the excess sillimanite had melted.

[9] *Ref. 33*, p. 487.


TRANS. AM. CER. SOC. VOL. XVIII
FIG. 4.
KIRKPATRICK
THEORETICAL BINARY SYSTEM
X
g
t_1
f
t_2
e
h
A
B
d
t_3
t_4
c
k
w
t_5
b
r
n
s
E
a
m
0 10 15 20 30 40 45 50 60 70 80 90 100
PERCENT B

In isomorphous systems, the melting point eutectic has no effect on the deformation points of other mixtures in the series.

Reactions and Chemical Nature of the Substances. Sosman,[10] in connection with the discussion just quoted, says:

> "The first and most important cause (for all the cones not going down at the eutectic temperature) is the fact that the silicate cones are not homogeneous in the chemical sense. Cones 28 to 41 are made up of mixtures of quartz and kaolin, or alumina and kaolin. The quartz must invert into cristobalite, and the kaolin must decompose into cristobalite and sillimanite. In cones 28 to 37, sillimanite and excess cristobalite must then melt together into the eutectic; in cones 38 to 41, cristobalite and excess alumina must unite to form more sillimanite, and this again melts down with excess alumina into a eutectic. The cones as made up have therefore not the same physical constitution that they would have if first melted and then made up out of the melted material. The second cause is the slow time-rate of melting of silica. The third reason is the very viscous character of the liquid which is formed."

The influence of the heat of formation of compounds on deformation points has already been mentioned.

In the systems studied in this investigation the most important reaction is the expulsion of CO_2 from calcite and magnesite. This is most rapid in the mixtures at from 1000° to 1200°. At 1200° and above, cones containing 60 percent or more calcite or magnesite could be seen to be very porous.

Other important reactions which probably occur and which will be discussed later are the decomposition and the formation of compounds.

Size, Shape and Inclination of Cone. An excellent and thorough study of the effect of a number of different factors on the deformation points of coal ash was made by Fieldner and Hall.[11] The average composition of the ashes tested was as follows:

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[11] *Ref. 48d.*
[10] *Ref. 33*, p. 487.

SiO_2	50
Al_2O_3	25
Fe_2O_3	12
TiO_2	1.4
CaO	26 samples average 2.7 percent 9 samples average 15.5 percent
MgO	1.6
Na_2O	0.9
K_2O	1.3
SO_2	2.3

As indicated by the analyses, coal ashes contain those oxides found in impure clays and in about the same proportions. Hence conditions which were found to give the most accurate determinations of deformation points of such complicated mixtures as coal ashes should give the most accurate results for many ceramic mixtures.

Fieldner and Hall conclude that:

"Triangular shaped cones ¼ inch base and 1¼ inches high give closer duplication and from 10° to 50° lower average softening points than ¼ inch by 1 inch cones (due to the greater bending moment of the former). Cones 3/16 inch by 1 inch gave practically the same results as ¼ inch by 1½ inch cones. The more slender cones were more satisfactory in giving shorter and more definite softening intervals; they also gave less trouble from intumescence due to evolution of gases from the melting ash. In reducing-atmosphere tests, the surrounding gases penetrated a thin cone more uniformly than one with a wide base."

"Mounting the cones with a considerable inclination, 35° to 45° from the vertical led in some cases to premature deformation points, which were caused by a further bending over, due to shrinkage of the cone in its base, rather than deformation due to softening and flowing of the ash. Vertical or nearly vertical cones were free from this source of error and gave the most concordant indications."

"Ash ground to an impalpable power tended to soften at a slightly lower temperature than 100 mesh ash. The difference averaged 6° and in no case exceeded 40°."

The materials must be mixed by some method which will give a physically homogeneous body. Segregation of different minerals would tend to cause higher or lower deformation points.

Fieldner and Hall conclude further:

"The atmosphere in which the ash was heated proved by far the most important factor in causing large variations in the softening temperature. The highest softening points were obtained in an atmosphere of air or in a strongly reducing atmosphere of CO, which prevented the iron oxide from acting as a fluxing agent by reducing it to metallic iron before the softening of the ash began. The lowest softening temperatures were obtained in those atmospheres of mixed gases in which reduction of Fe_2O_3 proceeded mainly to FeO, the most active phase of iron as regards slag formation at lower temperatures. The maximum variation in softening points due to the nature of the atmosphere ranges from 143° to 495°C."

Some of the Seger cones below No. 4 contain Fe_2O_3 or PbO as a flux. Under reducing conditions these deform, in general, at lower temperatures. Even those cones containing no iron may be "flashed down" if not protected from the kiln gases, giving indications one or two cones low.

Rate of Heating. Continuing the quotation of Fieldner and Hall's conclusions:

"In general, slower rates of heating gave lower softening points. Rates slower than 2° per minute are too time consuming for practical consideration. Rates faster than 10° per minute led to inaccurate temperature measurements. A 2° rate of heating gave the most uniform results: however, heating at 5° per minute to initial deformation and then reducing to 2° per minute, gave practically the same results and saved considerable time."

In discussing the behavior of Seger cones, Rieke[12] says:

"Many determinations in different kilns show that the melting points (deformation points) of all Seger cones 022-15 are affected more or less by the duration of heating. In contrast with this factor, all others influencing the melting-down of cones, appear of less importance. While the lower cones (022-012a) deform sooner with very slow heating than with quick heating, the cones from 012a to 1 with excessively slow heating become more refractory due to formation at their surfaces of a glaze of difficultly fusible compounds. The intervals between successive cones thus become greatly changed and in some cases greatly lessened, altho the cones go down in proper order. From about Seger cone 1 to cone 15, all cones deform at lower temperatures, with slower heating than with quick heating. This lowering can amount to from 60° to 100°."

[12] *Ref. 63*, p. 743.

Dr. Rothe of the Physikalische-Technische Reichsanstalt of Berlin conducted a very thorough investigation of the behavior of Seger cones[13] in which 400 observations were made on cones 4 to 20 in an iridium tube furnace.

He says:

"Since Seger cones are bad conductors of heat they will possess with quick heating a lower temperature than the thermo-element, which follows the temperature change quickly. We found by a series of experiments that the rate must be slowed up at least 20 minutes before the softening point is reached, to 5° at the most per minute. The cones were of small size and were inclined 10° from the vertical."

The work of Hoffman and of Rieke on Seger cones has been translated from the German by Geijsbeek. (*Trans. Amer. Cer. Soc.* XIV p. 849).

Simonis[14] determined deformation points of mixtures of Zettlitz kaolin, quartz and feldspar. He says:

"If we have compositions whose components show great tendency to reciprocal solution, formation of a definite chemical compound or whose melting-point lies far below that of the components, then the melting-point lies close to the deformation point, and the softening takes place quickly and at a more definite temperature."

"In bodies whose components show only slight reaction on one another and in which the cone melts somewhat higher than the lower component, softening is slow. In the ideal case no solution of the more refractory constituent occurs in the first melted glass and the cone goes down slowly. The softening rests only in part on the gradually resulting solution of the high melting components, in other part on the decrease in viscosity of the lower melting components.[15] Mixtures of the first kind are in general more independent of the rate of heating than those of the second kind. The rate of heating was on the average such that a five minute interval elapsed between the going down of successive Z-Cones[16] (that is 4° to 5° per minute). At least this rate was maintained for the latter period of heating. The speed of heating far below the deformation point naturally played a far less important role."

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[13] *Ref. 60.*
[14] *Ref. 59.*
[15] This is apparently the case in the Al_2O_3-SiO_2 system.
[16] Seger cones made using Zettlitz kaolin and having deformation points close to those of the Seger cones.

Since it will be necessary in this investigation to discuss the deformation temperatures of some of the Seger cones, the following tables of temperatures are given.

Temperatures given by the 1910 circular of the German Reichsanstalt:

CONE	TEMP. °C.	CONE	TEMP. °C.	CONE	TEMP. °C.
022	600	02a	1060	19	1520
021	650	01a	1080	20	1530
020	670	1a	1100	26	1580
019	690	2a	1120	27	1610
018	710	3a	1140	28	1630
017	730	4a	1160	29	1650
016	750	5a	1180	30	1670
015a	790	6a	1200	31	1690
014a	815	7	1230	32	1710
013a	835	8	1250	33	1730
012a	855	9	1280	34	1750
011a	880	10	1300	35	1770
010a	900	11	1320	36	1790
09a	920	12	1350	37	1825
08a	940	13	1380	38	1850
07a	960	14	1410	39	1880
06a	980	15	1435	40	1920
05a	1000	16	1460	41	1960
04a	1020	17	1480	42	2000
03a	1040	18	1500		

Rieke's table in *"Das Porzellan"* is the same as that given above except that he gives old cones instead of "*a*" cones as follows:

CONE	TEMP.	CONE	TEMP.
010	950	02	1090
09	970	01	1110
08	990	1	1130
07	1010	2	1150
06	1030	3	1170
05	1050	4	1190
04	1070	5	1200
03	1080	6	1210

In regard to the above scales, the following may be said. The figures given for cones 022 to 013a check closely those obtained by Rieke[17] at an average rate of 2° per minute. The temperatures for cones 013a to 6a were probably determined with the same rate of heating. Rieke[18] found cones 013a to 6a to deform about 45° higher at a rate of 4°-5° per minute. At a rate of 1° per minute, Rieke gets cones 7 to 15 down at from 10° to 25° lower than the Reichsanstalt figures. Hence the latter figures undoubtedly correspond to a rate of 2° per minute.

Dr. Rothe[19] using a rate of 5° per minute for 20 minutes before deformation, found that cones 7 to 13 deformed at from 55° to 15° degrees higher than the Reichsanstalt figures, and cones 14 to 20 deformed at temperatures close to these figures. The temperatures for cones 26 to 42 were obtained using a rate of 5° per minute.

Hence, it may be said that Seger cones under oxidizing conditions, may be caused to deform very close to the temperatures given in the above tables by the use of the following rates of heating:

Cones	*Rate*
022 to 13 inc.	2° per minute
015a to 6a inc.	2° per minute
14 to 42 inc.	5° per minute

In summarizing the observations quoted above in regard to the effect of rate of heating, the following points are noted: —

1. The rate of heating affects cones of different compositions differently. The principal reason is probably that the speeds of reactions tending to establish equilibrium conditions between liquid and solid phases change more rapidly with change of temperature in some mixtures than in others.

2. The less viscous the eutectic portions of the cones or the more liquid the entire mass, the less effect has the rate of heating.

3. Rieke found that some Seger cones melted lower and some higher at slow rates of heating.

[17] *Ref. 63*, p. 727.
[18] *Ref. 63*, p. 742.
[19] *Ref. 60.*

4. Fieldner and Hall very properly introduce the practical consideration that rates slower than 2° per minute are too time-consuming for use in laboratory tests.

5. The final test of the suitability of a rate of heating for any one composition or for a series of mixtures of similar properties is the degree of variation between deformation temperatures obtained by repeated determinations.

In the present investigation, check determinations were made in one series, orthoclase-calcite. The average difference from the mean value of deformation temperature was less than 2°, that is, within the accuracy of the pyrometer used. The rate used (4° per minute) therefore gave results sufficiently accurate for the purpose. At this rate, the deformation temperatures for Seger cones 3, 6, 9 and 10 were about 50° higher than the Reichsanstalt figures which correspond to a rate of 2° per minute. Stull and Howat,[20] using a rate of 2½° per minute for porcelain glaze cones, obtained about the same degree of accuracy.

General Statement Regarding Effect of Various Factors on Deformation of Cones. The above list of factors is not considered complete but is intended to give an idea of the great number of variables affecting such determinations and perhaps includes the most important. With so many possible factors affecting the deformation of cones, the degree of accuracy obtained in determination of deformation temperatures is remarkable. In addition to the separate effect of each factor is the effect of one factor upon others. For example, the effect of melting point eutectic depends upon its viscosity. The deformation temperature itself, however, is of doubtful value unless the conditions under which it was determined are given.

METHODS USED

The deformation tests in the present study were made in a platinum resistance furnace of one inch inside diameter using direct current at 110 volts. The rheosat used gave rather large steps in resistance so that it required a shifting adjustment in order to maintain the slow rate of temperature rise.

[20] *Ref. 37.*

The temperature measurements were made by means of a Heraeus platinum platinum-rhodium thermocouple and a Leeds Northrup indicator potentiometer accurate to about 2 millivolts (2°). The thermocouple was calibrated by the method described by Fulton,[21] using two points, the temperature of boiling sulphur and the melting point of copper, the cold junction being maintained at 0°C.

The minerals used were furnished by the Foote Mineral Co., of Philadelphia. They were crushed in a jaw crusher to about half-inch size. The material passing through a 20 mesh screen was rejected. The larger sized material after hand picking to remove impurities, was ground dry in porcelain ball mills using flint pebbles and passed through a 200 mesh screen.

The cone mixtures were made in five gram amounts by rolling the powders on paper, passing through a 100 mesh screen, then grinding in an agate mortar. Five percent dextrine solution was added, and cones molded in an Orton cone mold. This gave cones ¾ inch high and 3/16 inch side of base. These were found to give consistent results. There was not time to determine the effect of size, as done by Fieldner and Hall.[22]

The cones were set at an angle of 25° from the vertical in pats composed of 50 percent bauxite and 50 percent kaolin, two cones being placed in each pat. In the orthoclase-calcite series, the pats containing cones were not calcined before the deformation test was made. In all other series they were calcined to 800° in an electric furnace. The furnace in which the tests were made was brought to 800° after each determination, and the pats transferred to it from the calcining furnace at this temperature. The temperature was then raised at the rate of 10° per minute to a point at or below the beginning of deformation. This point could not be kept uniform because the temperature of deformation could not be closely predicted and there was not time for preliminary tests. From this point, the rate was maintained at 4° per minute to the end of the test. That this rate gave consistent results in nearly all cases is shown by the check results obtained in the orthoclase-calcite series. At

[21] *Ref. 48e.*
[22] *Ref. 48d.*

this rate, Orton cone 9 went down at 1340° (nominal temperature 1280°) and cone 10 at 1357° (nominal temperature 1300°).

The deformation temperature was taken as that temperature at which the cone bent and the top touched the upper surface of the pat or at which it fused to a ball.

ORTHOCLASE-CALCITE SYSTEM

Properties of Components. The microcline employed, when prepared for use, had the following composition:—

Percent Composition		Mineral Composition Calculated	
	percent		*percent*
SiO_2	66.00	Orthoclase	73.40
Al_2O_3	19.00	Albite	19.00
K_2O	12.34	Corundum	1.86
Na_2O	2.24	Quartz	4.92
Fe_2O_3	00		
TiO_2	00		99.18
Loss on Ignition	.25		
	99.83		

Ceramic formula:

$$\left.\begin{matrix}.7843\ K_2O \\ .2157\ Na_2O\end{matrix}\right\} 1.113\ Al_2O_3\ 6.571\ SiO_2$$

Mol. Weight 595.

The mineral composition is calculated according to the method of Cross.[23]

Determinations of the temperature range of melting of potash feldspars are given by Doelter[24] the average temperature of beginning of melting being 1170°, of complete melting 1205°, melting interval 35°. Potash feldspars deform with Seger cones 8 to 10, depending upon the amounts of albite, anorthite, alumina, quartz, etc., which they contain.

The deformation point of the orthoclase used in this investigation was the same as that of Seger cone 9, the rate of heat-

[23] *Ref.* 48, p. 186.
[24] *Ref.* 48a.

ing being 4° per minute and the temperature 1338°. Orthoclase exists in only one crystalline form and is stable at all temperatures. A very complete study of orthoclase and its properties is given by Doelter.[25]

The calcite employed had the following analysis when prepared for use:

Percent Composition		Mineral Composition Calculated	
	percent		*percent*
CaO	54.96	Calcite	98.0
MgO	1.02	Magnesite	2.0
SiO_2	0.69	Quartz	0.7
Al_2O_3, Fe_2O_3	.02		100.7
Na_2O	.18		
K_2O	.00		
CO_2 calculated	43.10		
	99.97		

Ceramic formula

$$\left.\begin{matrix} .9722\ CaO \\ .0250\ MgO \\ .0029\ Na_2O \end{matrix}\right\} \quad .002 \quad \left\{\begin{matrix} Al_2O_3 \\ Fe_2O_3 \end{matrix}\right. \left\{\begin{matrix} 0.98\ CO_2 \\ .0114\ SiO_2 \end{matrix}\right.$$

The melting point of CaO has been determined by Kanolt as 2570°.[26] CaO fuses to a clear liquid of low viscosity which crystallizes readily into a well developed cubic habit in the isometric system. Lime being itself quite liquid when melted, tends to cause sudden liquefaction in a mixture in which it is present in sufficient amount.

Results. The results for this system are shown in Fig. 5 and in Table I. The deformation point curve has two maximum points and three minimum points. The lag in the curve from 8 percent to 12 percent will be neglected since the components are not sufficiently pure to allow of a satisfactory explanation.

[25] *Ref. 48a*, II-9-1915.
[26] *Ref. 46*, p. 3.

Since the system is poly-component and microscopic examinations were not made, an attempted explanation of results consists necessarily of a statement of possibilities. If the components were pure, the maximum between 25 percent and 30 percent

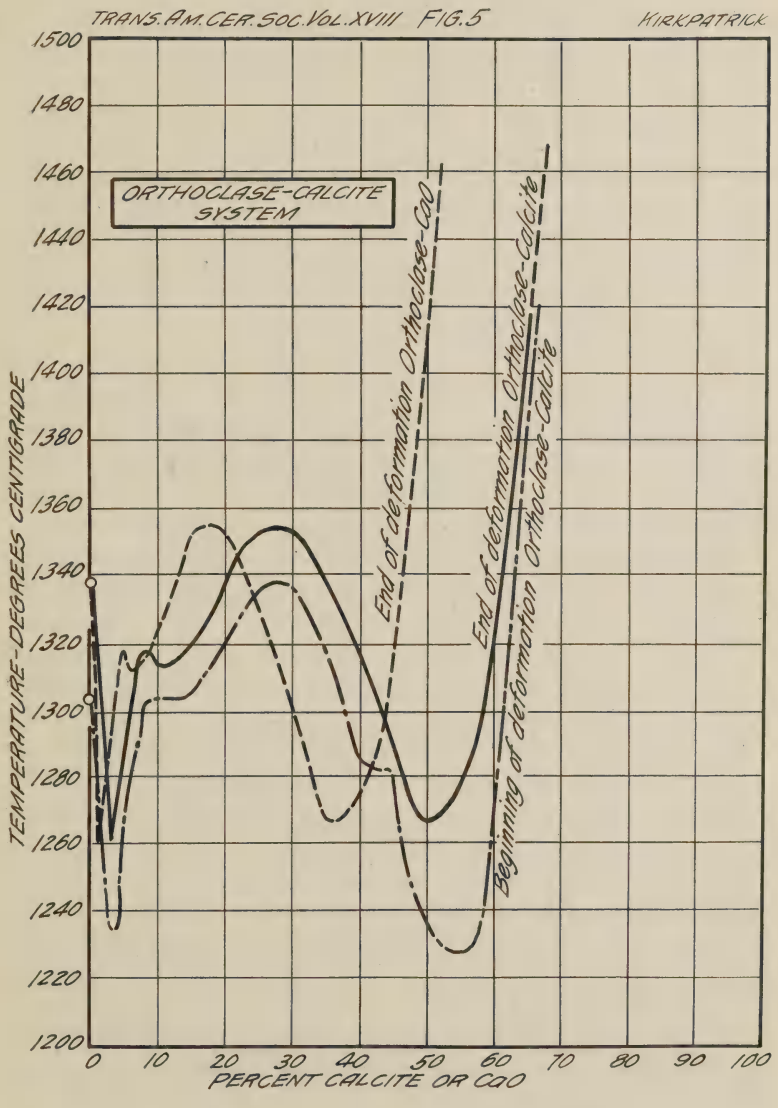
TRANS. AM. CER. SOC. VOL. XVIII TABLE I KIRKPATRICK

ORTHOCLASE-CALCITE SYSTEM

Percent Calcite	Rate reduced to 4° per min. at degrees centigrade	Deformation began degrees centigrade	Deformation ended degrees centigrade	Softening interval degrees centigrade	Appearance of cone of deformation temperature
0	1278	1303	1338	37	
1	1252	1282	1312	30	
2	1252	1260	1295	35	
3	1232	1234	1261	27	
4	1232	1234	1271	37	
5	1232	1261	1283	21	
6	1232	1283	1303	20	
8	1278	1303	1318	16	
10	1278	1303	1313	10	
12	1278	1303	1313	10	
14	1278	1303	1318	15	
17	1278	1310	1326	16	
20	1278	1320	1338	18	
25	1303	1336	1353	17	
30	1303	1336	1353	17	
35	1278	1320	1338	18	
40	1278	1285	1315	30	
45	1232	1282	1292	10	
50	1232	1236	1266	30	
55	1210	1227	1278	51	
60	1252	1272	1324	52	
65	1303	1392	1417	25	Dusted on cooling
70	1336	1440	—	—	Dusted on cooling

calcite would correspond closely to the formula 2CaO.Or[27] (26.4 percent calcite). The calculated mineral composition is: 2CaO, 0.75 Or, 0.21 Ab, 0.1 corundum, 0.5 quartz. It is doubtful whether such compounds as 2CaO.Or or 2CaO.Ab would form

[27] Or = orthoclase, Ab = albite.


TRANS. AM. CER. SOC. VOL. XVIII FIG. 5
KIRKPATRICK
ORTHOCLASE-CALCITE SYSTEM
End of deformation Orthoclase-CaO
End of deformation Orthoclase-Calcite
Beginning of deformation Orthoclase-Calcite
TEMPERATURE-DEGREES CENTIGRADE
1500
1480
1460
1440
1420
1400
1380
1360
1340
1320
1300
1280
1260
1240
1220
1200
0 10 20 30 40 50 60 70 80 90 100
PERCENT CALCITE OR CaO

since no natural compounds are known containing CaO and Or or Ab in the same molecule in these ratios.

If the maximum point does not represent the formation of a predominating compound it may be due to other reactions in the system.

Purdy[28] says,

> "There is no reason to believe that feldspar is affected chemically even in completely fused Bristol glaze mixtures unless there is not sufficient silica present to satisfy the calcium and zinc oxides, and further, that if the feldspar should be so affected it would be converted into leucite (K_2O Al_2O_3 $4SiO_2$) the pyro-properties of which are so like those of the orthoclase feldspar as to make but little difference in the behavior of the resultant fusion."

Calculations show that in mixtures from 0 to 25 percent calcite, the CaO is sufficiently satisfied with the excess Al_2O_3 and SiO_2 in the orthoclase so that little or no decomposition of feldspar would take place; while beyond 25 percent calcite, the CaO is not thus satisfied and probably does attack the orthoclase.

There is little doubt as to decomposition of alkali feldspars occurring in the presence of sufficient lime at high temperatures. Chemists claim that the feldspar in limestone is decomposed by the lime on heating over a Bunsen burner, and use this method of decomposition in limestone analysis. Doelter[29] describes a number of processes in which lime is used alone or in mixtures added to feldspar for the purpose of decomposing the latter into alkali, calcium aluminium silicates, calcium silicates, etc. The various processes had in view the use of the products as fertilizer and as Portland cement. At the temperatures used in the present investigation, 1150° to 1475° decomposition of the alkali feldspars very probably occurs. The extent of this decomposition and the compounds formed could have been determined only by chemical and microscopic examinations. These were beyond the scope of the present work.

That part of the curve between 0 and 25 percent calcite may be explained as follows: The orthoclase-albite mixture is considered unacted upon chemically within this range of composition.

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[28] *Ref.* 24, p. 96-97.
[29] *Ref.* 48*a*, II-9, p. 569.

The remainder of the mixture is composed of Al_2O_3, SiO_2 and CaO. If the percentages of these oxides are calculated in a number of mixtures the following results are obtained.

MIXTURE PERCENT CALCITE	DEFOR. TEMP.	PERCENT OF TOTAL MIXTURE	OXIDE MIXTURE SiO_2:Al_2O_3:CaO			MELTING POINT[30]	MELTING POINT OF TERNARY EUTECTIC
2	1295°	7.8	62.2	23.5	14.3	1415°	Point 2 — 1165°
3	1261	8.2	57.7	21.9	20.4	1270	Point 2 — 1165
8	1318	11.8	38.4	14.5	47.1	1400	Point 6 — 1310
25	1353	19.1	19.3	7.3	73.4	2000	Point 17 — 1470

As soon as the ternary eutectic in any of these mixtures melts it will tend to cause the cone to deform. The 3 percent calcite orthoclase mixture contains the oxides SiO_2, Al_2O_3, and CaO in a lower melting proportion than any other of the above mixtures. Hanna[31] found the mixture of Rankin and Wright's eutectic point 2 to deform at cone 5-1/3, about 1200°, or 35° above its melting point. Our 3 percent mixture deformed at 1261°, corresponding to 1210° if rate of heating were 2° per minute. The shape of the deformation point curve from 0 to 25 percent calcite is due then to the addition to the orthoclase used, of mixtures of greater or less degrees of fusibility. Since most of the commercial orthoclases contain excess Al_2O_3 and SiO_2, they would probably give a eutectic with calcite at low percentages of the latter as well as near 50 percent.

It may be thought that the decrease in deformation point up to 3 percent calcite is not due to the reaction between Al_2O_3, SiO_2 and CaO but is the natural eutectic between orthoclase and calcite. Consideration of the behavior of lime-silica mixtures shows that this is improbable. Silica and the alkali feldspars, near their melting points, possess about the same degree of viscosity, and the action of lime in small amounts should be similar in both cases. The lime-silica deformation point curve (Fig. 1) slopes gradually from SiO_2 to 20 percent CaO (35.7 percent calcite), the deformation point lowering being 90°, while in our orthoclase-calcite

[30] *Ref. 46.*
[31] *Ref. 39*, p. 683.

system a lowering of 77° was obtained with 1.68 percent CaO (3 percent calcite). In view of these facts it seems improbable that the 3 percent eutectic is the real eutectic between orthoclase and calcite.

Beyond 25 percent calcite, the CaO is not satisfied with free Al_2O_3 and SiO_2 and decomposition of feldspar probably takes place. Possible reactions are as follows:

1. $KAlSi_3O_8 + CaO \rightarrow KAlSi_2O_6 + CaSiO_3$.
   (orthoclase) (leucite)
2. $2KAlSi_3O_8 + 3CaO \rightarrow 2KAlSi_2O_6 + Ca_3Si_2O_7$.
3. $KAlSi_3O_8 + 2CaO \rightarrow KAlSi_2O_6 + Ca_2SiO_4$.
4. $KAlSi_3O_8 + 3CaO \rightarrow KAlSi_2O_6 + Ca_3SiO_5$.
5. $KAlSi_2O_6 + CaO \rightarrow KAlSiO_4 + CaSiO_3$.
   (leucite) (potash nephelite)
6. $2KAlSi_2O_6 + 3CaO \rightarrow 2KAlSiO_4 + Ca_3Si_2O_7$.
7. $KAlSi_2O_6 + 2CaO \rightarrow KAlSiO_4 + Ca_2SiO_4$.
8. $KAlSi_2O_6 + 3CaO \rightarrow KAlSiO_4 + Ca_3SiO_5$.
9. $KAlSiO_4 + CaO \rightarrow KAlO_2 + CaSiO_3$, etc.
10. $NaAlSi_3O_8 + 2CaO \rightarrow NaAlSiO_4 + 2CaSiO_3$, etc.
    (nephelite)
11. Reactions between the above products.

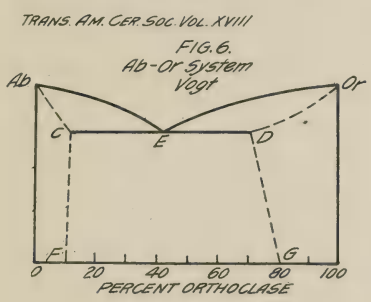
Some of these reactions may be ionic, causing the system to be still more complicated.

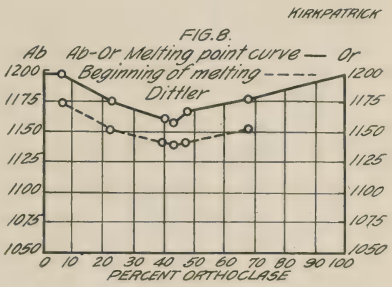
The lowering in deformation point from 25 percent to 50 percent calcite would then be due to eutectics in systems containing varying amounts of orthoclase, leucite, albite, nephelite, calcium silicates and other compounds. Also, the heat of formation of these silicates might aid in causing lower deformation points. Beyond 50 percent, formation of the orthosilicate and finally excess of free lime would cause the curve to rise abruptly. The cones containing 65 percent and 70 percent calcite dusted immediately on cooling, indicating the probable presence of the orthosilicate. The dusting could hardly have been due to hydration of CaO since this is a very slow process for lime calcined to such high temperaturees (1420°-1440°).

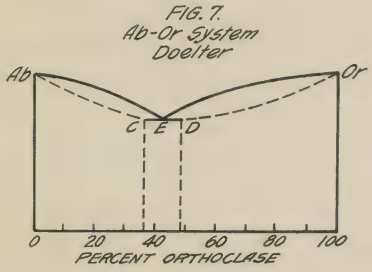
If the components had been pure, it is probable that decomposition of felspar would have begun with low percentages

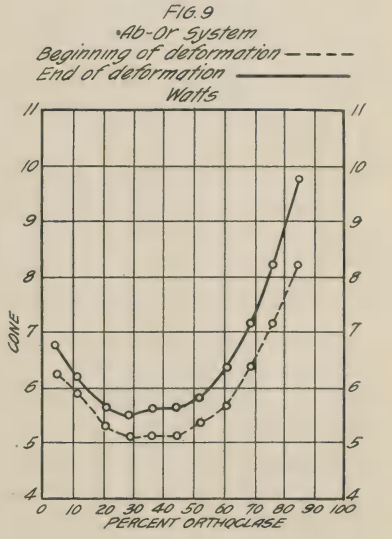
of calcite causing the deformation point curve to slope gradually to the 50 percent eutectic.

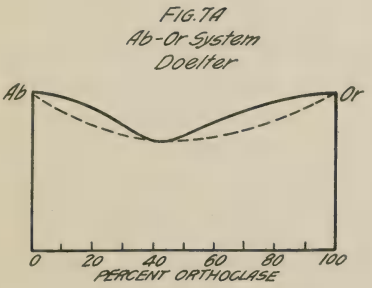
The above interpretation seems to fit the observed phenomena fairly well. However, the system should be discussed with











another possibility in mind, namely, that decomposition of feldspar does not occur and that the 50 percent eutectic is a natural eutectic between orthoclase, albite and CaO.

The system orthoclase-albite is an isomorphous eutectic

series. This has been determined by the examination of natural minerals which were formed under conditions that cannot be duplicated in the laboratory. In the heating of ceramic mixtures, the alkali feldspars are always obtained in the glassy condition, but a knowledge of the nature of the crystallization or melting point curves for such systems aids in understanding the behavior of these minerals in ceramic bodies.

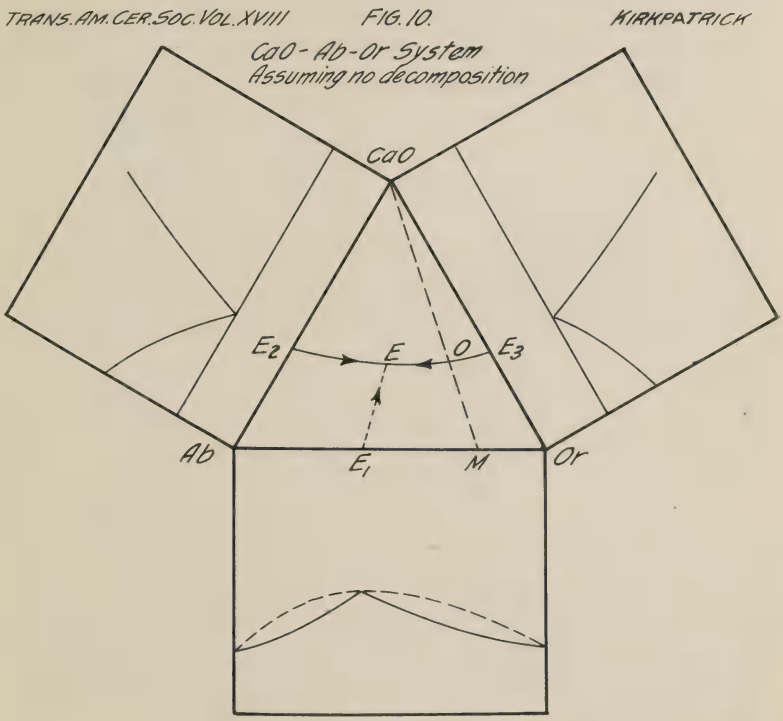
Vogt[32] assigns the above system to Roozeboom's[33] type V as shown in Fig. 6. Doelter[34] and Dittler[35] think that the mixed crystal gap CD is very small as shown in Fig. 7 or entirely absent as in Fig. 7A. The melting point curve for this system, determined by Dittler using the heat-microscope[36] is shown in Fig. 8. The determinations were made upon the natural minerals, using a rate of heating of 5° per minute from 20° to 1000° and 2½° per minute above 1000°. He concludes that orthoclase and albite have nearly the same melting temperatures. In Fig. 9, Watts' data on this system has been plotted from his Fig. 1.[37] His deformation eutectic range corresponds to the melting point eutectic found by Dittler.

When calcite in amounts above 30 percent is added to the orthoclase used in this investigation, the mixtures consist, mainly (assuming no decomposition) of orthoclase, albite and calcite, a ternary system with small amounts of inert SiO_2, Al_2O_3 and MgO. The systems CaO-Or. and CaO-Ab. would probably be systems of the simple type shown in Fig. 4. Hence the ternary melting point diagram would be of the form shown in Fig. 10. Discussions of the probable nature of such systems are given by Vogt[38] and by Bowen.[39] The deformation point diagram, using pure components would resemble the melting point diagram. Mixtures in our orthoclase-calcite system would be represented by points on the line M-CaO, neglecting impurities, and our 50 percent eutectic would be located at some

[32] *Ref. 48a*, I-5, p. 782.
[33] *Ref. 48a*, I-5, p. 772.
[34] *Ref. 48a*, II-9, p. 482.
[35] *Ref. 48g.*
[36] *Ref. 48a*, I-5, p. 646.
[37] *Ref. 29*, p. 153.
[38] *Ref. 48f*, p. 523.
[39] *Ref. 78*, p. 220.

point within the triangle as O. Beyond this, increase in CaO would cause a rapid rise in deformation temperatures.

The only effect of the albite content of the orthoclase would be to lower the deformation point of the 50 percent calcite mixture perhaps 20°, hence, practically the system might be treated as binary with the components, calcite: Or. + Ab, in the same manner as Vogt[40] treats the system Or.: Ab + An.



The above explanation, in which no decomposition of feldspar is assumed, does not seem to afford as satisfactory an interpretation of the observed phenomena as does the previous one, assuming decomposition.

Seger[41] attempted to determine a eutectic between feldspar and calcite. He used only three mixtures:

Or. $CaCO_3$ .....15.2 percent $CaCO_3$,
Or. $2CaCO_3$ .....26.4 percent $CaCO_3$,
Or. $3CaCO_3$ .....35.0 percent $CaCO_3$,

and found them to be no less fusible than feldspar. Examination of the curve, Fig. 5, shows that Seger missed the eutectic mixtures and that the feldspar he used was probably more pure than that used in this investigation.

The 3 percent eutectic checks that obtained by Bleininger.[42] His materials are compared with those used in this investigation as follows:

	Our Orthoclase	*Bleininger's Feldspar*
Or	73.40	64.60
Ab	19.00	14.50
An		4.56
Corundum	1.86	1.52
Quartz	4.92	14.90
$MgO\ SiO_2$		.04

	Our Calcite	*Bleininger's Calcite*
$CaCO_3$	98.0	94.0
Al_2O_3		3.5
SiO_2	0.7	1.6
$MgCO_3$	2.0	

It is seen that the impurities in the mixtures used by Bleininger would give Al_2O_3-SiO_2-CaO proportions similar to those in our mixtures, hence his curve should be similar to that of Fig. 5.

The minimum point at 30 percent calcite has the following ceramic formula:

$$\left.\begin{matrix} .661\ K_2O \\ .186\ Na_2O \\ .150\ CaO \\ .0036\ MgO \end{matrix}\right\} 0.937\ Al_2O_3\ 5.54\ SiO_2,$$

[40] *Ref. 48f.*
[41] *Ref. 44,* p. 232.
[42] *Ref.* 11, *p.* 263.

that at 50 percent,

$$\left.\begin{array}{l} .114\ K_2O \\ .034\ Na_2O \\ .831\ CaO \\ .0214\ MgO \end{array}\right\}\ 0.161\ Al_2O_3\ 0.962\ SiO_2$$

The appearance of the cones in this series indicated increased surface tension from 17 percent to 60 percent calcite causing balling-up, while other mixtures deformed normally. The softening intervals do not change consistently with the deformation temperatures, indicating that the magnitude of the interval is governed by several factors. All cones which went down were glossy in appearance.

Applications of Results. Eutectic mixtures of ceramic materials have been used for various purposes. The best porcelain and Bristol type glazes lie in eutectic areas. This is shown by Stull for porcelain glazes[43] and Watts for Bristol glazes.[44] Seger used what he supposed to be the most fusible mixture of feldspar, whiting, clay and flint as starting point for his cone series, that is, cone 4. Hanna[45] found a mixture of these substances which deformed at cone 2¾.

The eutectic between feldspar and talc has been used by the Bureau of Standards[46] as a flux to increase the strength of porcelain bodies.

Several investigations have been conducted to determine the effect of replacing feldspar by $CaCO_3$ in ceramic bodies. Ashley[47] states that whiting added to an earthenware body in amounts not to exceed 3 percent, increased shrinkage, decreased porosity, increased translucency and very decidedly raised the strength (rattler test). The ratios $\frac{CaCO_3}{CaCO_3 + \text{Feldspar}}$ were 1.6 percent, 4.7 percent, 11 percent, and 27.3 percent.

[43] *Ref. 87.*
[44] *Ref. 44.*
[45] *Ref. 39.*
[46] *Ref. 40.*
[47] *Ref. 13.*

Watts[48] found that in general, porcelain bodies containing whiting and feldspar in the proportion 4.3 percent whiting gave greater tensile strength than those containing more whiting.

It is possible that our eutectic mixtures containing 3 percent and 50 percent calcite may be added to bodies as fluxes, since they would tend to start vitrification earlier and give greater maturing ranges.

ORTHOCLASE-MAGNESITE SYSTEM

Properties of Components. The orthoclase was the same as that used in the previous series.

The magnesite used had the following composition:

Percent Composition		Mineral Composition	
	percent		*percent*
SiO_2	.00	Magnesite	95.5
Fe_2O_3, Al_2O_3	.56	Calcite	2.7
CaO	1.58	Al_2O_3	.54
MgO	46.78	K_2CO_3	.43
K_2O	.30	Na_2CO_3	.65
Na_2O	.66		99.82
CO_2	50.32		
	100.20		

Ceramic formula.

$$\left.\begin{matrix} .966\ MgO \\ .0235\ CaO \\ .0026\ K_2O \\ .0088\ Na_2O \end{matrix}\right\} .0046\ Al_2O_3 \left\{\begin{matrix} .0\ SiO_2 \\ .95\ CO_2 \end{matrix}\right.$$

The melting point of MgO has been determined by Kanolt[49] as 2850°. Crystals of the oxide from pure melt in the electric arc or from mixtures with $MgCl_2$ and SiO_2 are octahedral in habit with occasional cube faces, and are isotropic.

Results. The results for this system are shown in Table II. The deformation point curve, Fig 11, contains one minimum

[48] *Ref. 13.*
[49] *Ref. 48b.*

point and no maximum points. The eutectic at 3½ percent magnesite lies at a temperature 10° higher than the 3 percent eutectic in the orthoclase calcite system. This indicates that that portion of the mixtures composed of the ternary system MgO-SiO_2-Al_2O_3 has almost as much effect on the deformation points as the CaO-SiO_2-Al_2O_3 mixtures. If no impurities were present, it is doubtful if there would be a deformation eutectic between orthoclase and magnesite.

If the eutectic in the orthoclase-calcite system at 50 percent calcite (36 percent CaO) were a natural eutectic, there should be a similar eutectic in the orthoclase-magnesite system, since MgO in most systems tends to produce the same effect as CaO but to less degree. It would then appear that calcite was able to decompose the feldspar while magnesite was not.

TABLE II. ORTHOCLASE-MAGNESITE SYSTEM

PERCENT MAGNESITE	RATE REDUCED TO 4° PER MIN. AT °C.	DEFORMATION BEGAN °C.	DEFORMATION ENDED °C.	SOFTENING INTERVAL °C.
0	....	....	1338	35
1	1252	1278	1303	25
2	1252	1261	1288	27
3	1185	1232	1272	40
3.5	1185	1232	1271	39
4	1185	1232	1272	40
5	1185	1232	1296	64
10	1185	1315	1330	15
15	1252	1345	1353	8
20	1252	1345	1361	16
25	1303	1361	1374	13
30	1303	1361	1390	29
35	1303	1394	1422	28
40	1303	1411	1452	41
45	1303	1435	*	*
50	1303	1450	*	*
55	1303	....	*	*
65	1303	....	*	*

* Too high for furnace.

The ceramic formula of the 3½ percent eutectic mixture is as follows:

$$\left.\begin{matrix} .625\ K_2O \\ .174\ Na_2O \\ .196\ MgO \\ .005\ CaO \end{matrix}\right\} .886\ Al_2O_3\ \ 5.23\ SiO_2$$

All cones in this series deformed normally, indicating less surface tension than in the orthoclase-calcite system. All which went down were glossy in appearance. No previous work on this system is recorded.

Applications of Results. What has been said in this connection in regard to the orthoclase-calcite system applies here. MgO is used to some extent to replace feldspar in bodies. In small amounts it increases toughness and strength and improves color. In Germany, it is used in electrical porcelains where exact shapes are required. MgO is used to a considerable extent in glazes, replacing CaO and other oxides.

ALBITE-CALCITE SYSTEM

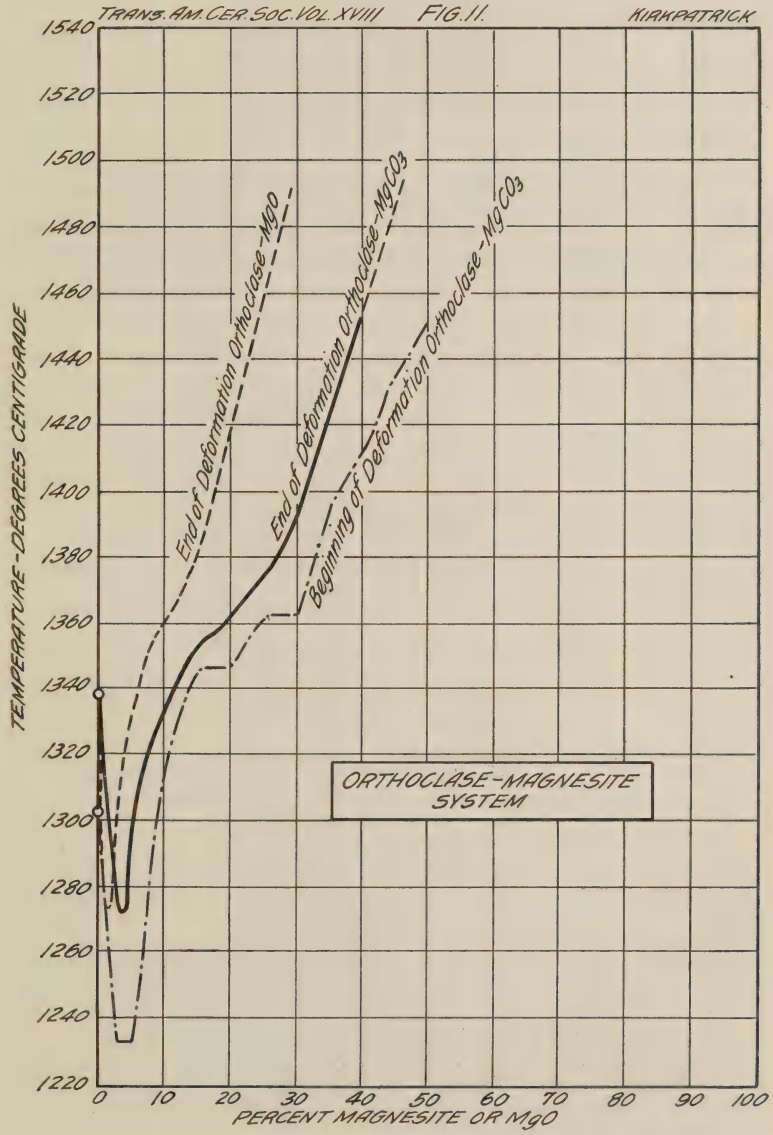
Properties of Components. The albite used had the following composition:—

Percent Composition	*percent*	Mineral Composition	*percent*
SiO_2	66.00	Albite	60.60
Al_2O_3	22.04	Orthoclase	5.92
CaO	2.98	Anorthite	14.78
MgO	0.74	Enstatite	1.83
K_2O	1.00	Corundum	3.79
Na_2O	7.14	Quartz	13.23
Loss on ignition	0.26		
	100.16		100.15

Ceramic formula.

$$\left.\begin{matrix} .584\ Na_2O \\ .054\ K_2O \\ .270\ CaO \\ .093\ MgO \end{matrix}\right\} 1.10\ Al_2O_3\ \ 5.58\ SiO_2$$

Mol. weight..... 552.


TRANS. AM. CER. SOC. VOL. XVIII
FIG. 11.
KIRKPATRICK
TEMPERATURE - DEGREES CENTIGRADE
1540
1520
1500
1480
1460
1440
1420
1400
1380
1360
1340
1320
1300
1280
1260
1240
1220
End of Deformation Orthoclase - MgO
End of Deformation Orthoclase - MgCO3
Beginning of Deformation Orthoclase - MgCO3
ORTHOCLASE - MAGNESITE SYSTEM
0
10
20
30
40
50
60
70
80
90
100
PERCENT MAGNESITE OR MgO

The albite was furnished as a pure mineral but the analysis shows it to be very impure.

Albite under heat treatment acts very much like orthoclase. Doelter[50] gives the melting range of Schmirn albite as 1135°

TRANS. AM. CER. SOC. VOL. XVIII TABLE III KIRKPATRICK

ALBITE-CALCITE SYSTEM

Percent calcite	Rate reduced to 4° per min. at degrees centigrade	Deformation began degrees centigrade	Deformation ended degrees centigrade	Softening interval degrees centigrade	Appearance of cone of deformation temperature
0	1236	.1236	1263	27	
	1185	1232	1260	28	
		Average	1262	28	
1	1185	1232	1250	18	
2	1142	1215	1240	25	
3	1142	1206	1223	17	
4	1142	1192	1223	31	
5	1150	1175	1210	35	
7.5	1150	1159	1183	24	
10.	1142	1155	1170	15	
15	1142	1146	1165	19	
20	1125	1151	1170	19	
25	1134	1134	1157	23	
30	1134	1134	1164	30	
35	1125	1159	1181	22	
40	1125	1164	1183	19	
45	1150	1180	1189	9	
50	1150	1189	1195	6	
55	1150	1186	1195	9	
60	1125	1236	1241	5	
65	1336	1360	1365	5	
70	1435	1435	1439	4	

to 1215°. Bowen[51] determined the melting point of pure albite to be 1100° ± 10°.

Commercial albites deform with Seger cones 5 to 7. The albite used in this series deformed in the interval from 1234° to 1262° with Seger cone 6 (rate of heating 4° per minute).

[50] *Ref.* 48*a*, I-5, p. 656.
[51] *Ref.* 75, p. 582.

The calcite used was the same as that in the orthoclase-calcite system.

Results. The results for this system are shown in Table III and in Fig. 12. The deformation point curve possesses a long eutectic range with the lowest point at 1157° and 25 percent calcite. The system has a high anorthite and free quartz content and hence differs considerably from the orthoclase-calcite system. Up to about 40 percent calcite, the lime is satisfied with silica and alumina, forming probably calcium aluminium silicates and calcium silicates. These would cause lowering of deformation points by eutectic formation. Beyond 40 percent it is probable that decomposition of albite and orthoclase takes place with the formation of nephelite, leucite and calcium silicates. Beyond 55 percent the amount of orthosilicate and CaO present in the mixtures becomes great enough to cause the curve to rise abruptly. The cones containing 65 percent and 70 percent calcite dusted quickly on cooling indicating the probable presence of calcium orthosilicate.

The ceramic formula for the 25 percent eutectic is as follows:

$$\left.\begin{matrix} .1785\ Na_2O \\ .1625\ K_2O \\ .6175\ CaO \\ .0418\ MgO \end{matrix}\right\} .331\ Al_2O_3\ 1.697\ SiO_2$$

Its deformation temperature was 1157° at rate of heating 4° per minute which corresponds to about 1110° at a rate of 2° per minute, or cone 01; this is a more fusible mixture than that obtained by Hanna.[52]

All the cones in the eutectic range balled up more or less, indicating high surface tension. Cones from albite to 30 percent calcite, inclusive, were glossy, those from 35 percent to 50 percent had a mat texture, the remainder were stony in appearance. The softening intervals are about of the same order as in previous systems, except that cones of from 45 percent to 70 percent calcite possessed very short intervals, 4° to 9° or 1 to 2 minutes.

[52] *Ref. 39*, p. 688.

Application of Results. Albite is used to some extent in porcelains. Radcliffe[53] found that albite gave better electrical porcelains than did orthoclase. Watts[54] found that albite porcelain gave a dead ring on striking, while orthoclase porcelain gave a clear ring; but both porcelains stood quenching tests and high voltage tests equally well. He found also[55] that albite gave to the porcelain matrix a remarkable dissolving power, causing a higher stage of solution at cone 10 than does orthoclase at cone 12. Some of the differences in properties of the two feldspars may be due to the nature of the impurities. Both always contain excess Al_2O_3 and SiO_2 while albite generally contains more anorthite than does orthoclase.

By adding lime to an albite of composition approaching that which we used, a mixture could be obtained which would begin softening action in a body 100° lower than would albite alone.

No previous work on this system is recorded.

ALBITE-MAGNESITE SYSTEM

Properties of Components. The albite and magnesite were the same as those used in the previous series.

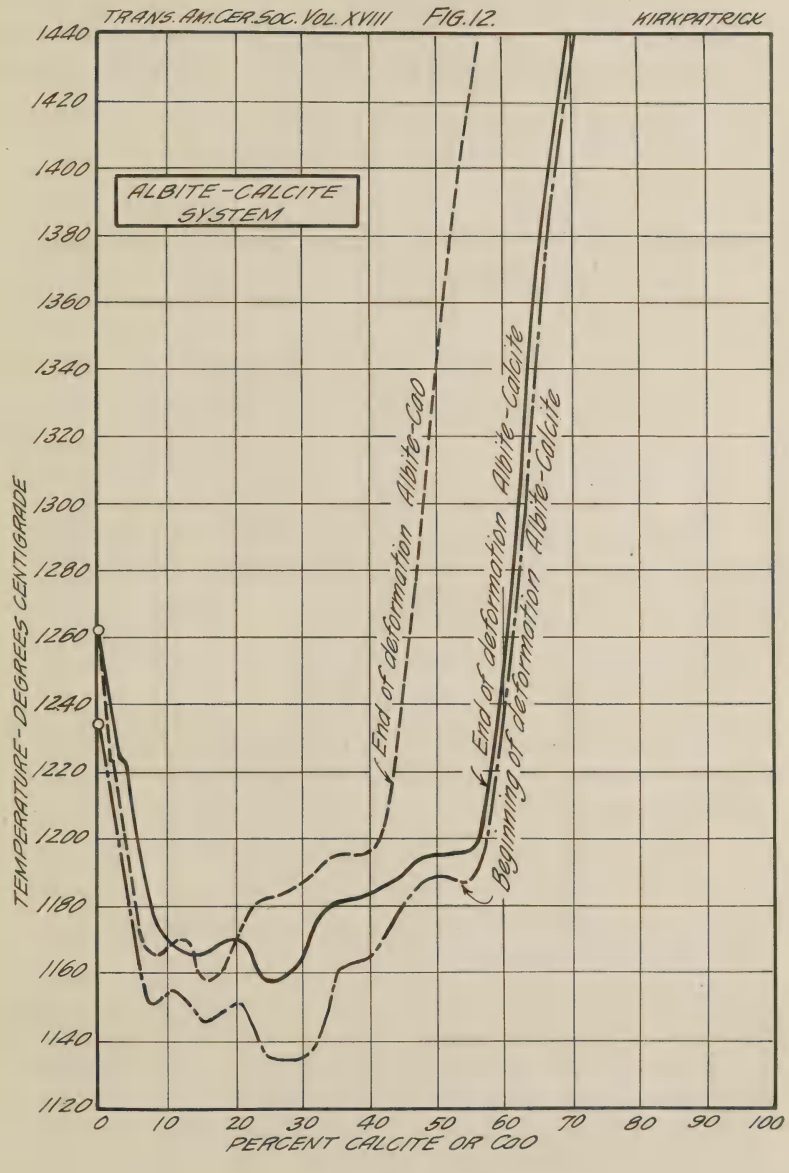
Results. The results for this system are shown in Table IV. The deformation point curve, Fig. 13, possesses a eutectic range from 5 percent to 20 percent magnesite. This is a shorter range and at a higher temperature than in the albite-calcite system, Fig. 12. It is a longer range at a higher temperature than in the orthoclase-magnesite system, Fig. 11. Reactions causing the eutectic are similar to those occurring in the albite-calcite system, but the magnesium compounds are less active.

The formula of the 15 percent eutectic is as follows:

$$\left.\begin{matrix} .2238\ Na_2O \\ .2033\ K_2O \\ .1119\ CaO \\ .4605\ MgO \end{matrix}\right\} .4385\ Al_2O_3\ \ 2.11\ SiO_2$$

$$.9995$$

[53] *Ref. 26.*
[54] *Ref. 15.*
[55] *Ref. 16*, p. 198.


TRANS. AM. CER. SOC. VOL. XVIII FIG. 12. KIRKPATRICK
ALBITE-CALCITE SYSTEM
End of deformation Albite-CaO
End of deformation Albite-Calcite
Beginning of deformation Albite-Calcite
TEMPERATURE-DEGREES CENTIGRADE
1440
1420
1400
1380
1360
1340
1320
1300
1280
1260
1240
1220
1200
1180
1160
1140
1120
0 10 20 30 40 50 60 70 80 90 100
PERCENT CALCITE OR CaO

This has a deformation point of cone 1¼, rate of heating 2° per minute.

The cones deformed normally and all were glossy in appearance.

As in the previous systems, the albite-magnesite eutectic mixtures could be added to other bodies as fluxes.

TABLE IV. ALBITE-MAGNESITE SYSTEM

PERCENT MAGNESITE	RATE REDUCED TO 4° PER MIN. AT °C.	DEFORMATION BEGAN °C.	DEFORMATION ENDED °C.	SOFTENING INTERVAL
0	....	....	1262	28
5	1125	1176	1193	17
10	1125	1168	1185	17
15	1125	1168	1185	17
20	1125	1168	1186	18
25	1125	1185	1205	20
30	1125	1210	1234	24
35	1252	1261	1283	22
40	1252	1286	1315	29
45	1294	1320	1351	31
50	1294	1345	1385	40
55	1378	1394	1431	37
60	1378	1460	*	*

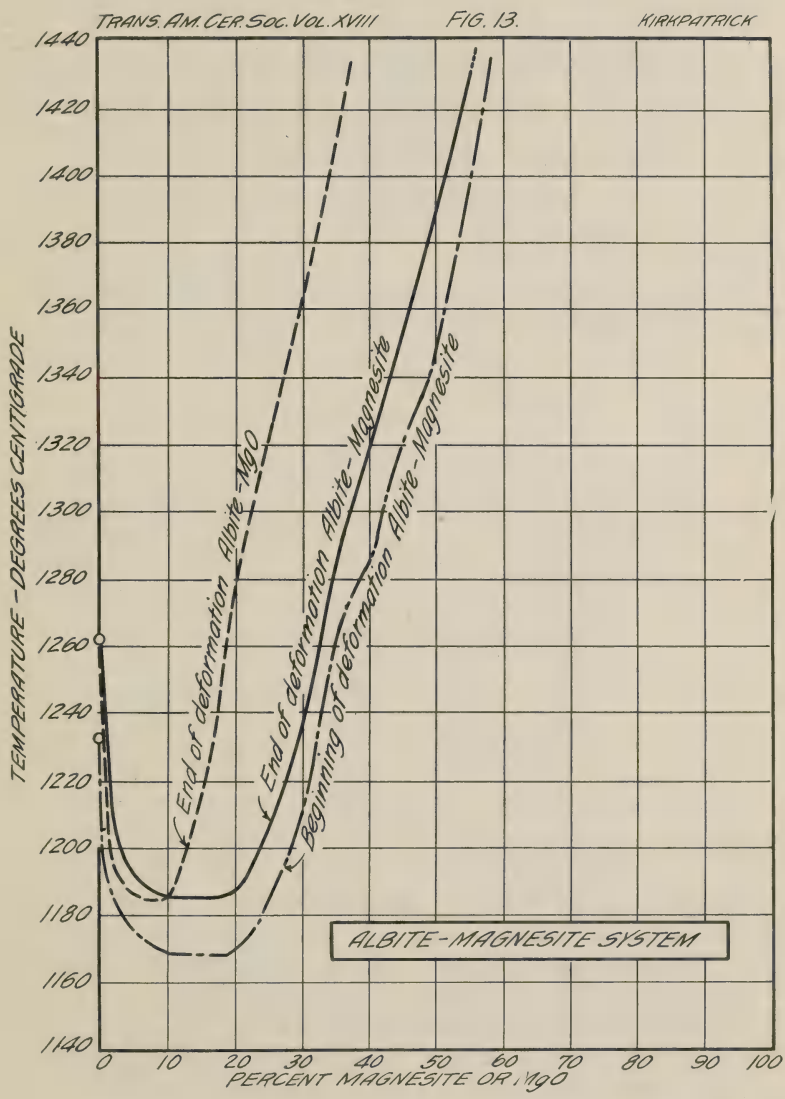
* Still up at 1476°.

SUMMARY

Deformation point-temperature curves have been determined for the following mineral systems, the components being somewhat impure:

1. Orthoclase-calcite,
2. Orthoclase-magnesite,
3. Albite-calcite,
4. Albite-magnesite.

System 1 possesses one well defined deformation point maximum at 27½ percent calcite and two well defined deformation point eutectics at 3 percent and 50 percent. There is probably no predominating compound formed in the series, which as has been shown is poly-component. The nature of the deformation point curve is probably due to the formation of several compounds and to the presence of eutectics between several components.


TRANS. AM. CER. SOC. VOL. XVIII
FIG. 13.
KIRKPATRICK
TEMPERATURE - DEGREES CENTIGRADE
1440
1420
1400
1380
1360
1340
1320
1300
1280
1260
1240
1220
1200
1180
1160
1140
End of deformation Albite - MgO
End of deformation Albite - Magnesite
Beginning of deformation Albite - Magnesite
ALBITE - MAGNESITE SYSTEM
0
10
20
30
40
50
60
70
80
90
100
PERCENT MAGNESITE OR MgO

System 2 has one deformation point eutectic at 3 percent magnesite, beyond which point the curve rises rapidly with increase of magnesite.

System 3: The albite used in this system was quite impure making the series poly-component. It possesses a long eutectic range at from 10 percent to 55 percent calcite, beyond which the curve rises rapidly with increasing calcite.

System 4 resembles system 3 in having a long eutectic range at from 5 percent to 20 percent, the action of magnesite being less than that of calcite.

The two lowest fusing mixtures obtained were as follows:

Albite-calcite system, 25 percent calcite.

$$\left.\begin{matrix} .1785\ Na_2O \\ .1626\ K_2O \\ .6175\ CaO \\ .0418\ MgO \end{matrix}\right\} \cdot 331\ Al_2O_3 \quad 1.697\ SiO_2$$

Deformation point cone 01 calculated for a rate of 2° per min.

Albite-magnesite system, 15 percent magnesite.

$$\left.\begin{matrix} .2238\ Na_2O \\ .2033\ K_2O \\ .1119\ CaO \\ .4605\ MgO \end{matrix}\right\} .4385\ Al_2O_3 \quad 2.11\ SiO_2$$

Deformation point cone 1¼ calculated for a rate of 2° per min.

The softening intervals of the mixtures, in general, bear no relation to compositions, or to deformation temperature, indicating that the viscosity range is governed by several factors.

The results tend to show that orthoclase and albite are decomposed by CaO, but are not decomposed by MgO.

An attempt has been made to explain the behavior of the various systems by means of the calculated mineral analyses of the components and of the known laws of eutectic formation.

The fact has been emphasized that in the determination of the deformation points of ceramic mixtures, especially of systems

containing commercial feldspar (which are known to be polycomponent), conclusions as to the causes of the observed phenomena should be made, keeping always in mind the actual compositions of the components, rather than assuming that the impurities have little effect.

The possible applications of the results are as follows:

1. The use of eutectic mixtures as fluxes in bodies and glazes.

2. The interpretation of fusion phenomena in mixtures containing the minerals used.

In conclusion, the writer wishes to acknowledge his indebtedness to Prof. A. V. Bleininger and Mr. R. K. Hursh for valuable suggestions in connection with the work.

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THE PRODUCTION OF CHINA FROM DOMESTIC MATERIALS

BY CHARLES F. BINNS AND GEORGE BLUMENTHAL

Following the study presented last year upon a characteristic of some American clays, there was undertaken in the laboratories at Alfred a series of experiments which had in view the solution of the problem of the production of china from the materials natural to the United States. This refers to the body generally known as American china or hotel china. It has no reference to either English bone china or to European porcelain.

The body mixture used was that given in the Government report on the pottery industry as a typical china:

Kaolin	27
Ball clay	15
Flint	36
Feldspar	19.5
Whiting	1.5
Heavy magnesium carbonate	1.0

The kaolin was the only ingredient which was varied. The ball clay was Johnson Porter, No. 11. The flint and spar were from the Eureka Co. of Trenton, N. J.

The kaolins were as follows:

A. English kaolin—MGR—1.
B. German kaolin of unknown origin.
D. Kaolin from Newark Kaolin Co., Delaware
D. Kaolin from Newark Kaolin Co., Delaware.
E. N. C. kaolin from Dillsboro.
F. N. C. kaolin from Sant and Son.
G. Georgia clay.
H. Florida clay.

Three mixtures were made up of each clay. Number 1, the whole mixture was blunged and lawned through 100 mesh. No 2, the ground material ground, blunged with the clays and lawned through 150 mesh. No. 3, the entire mixture was ground and lawned through 150 mesh.

Trial pieces were cast, burned to cone 10 in a commercial kiln and glazed. The results were sufficient to show that nothing could be expected from mere blunging. This at first seemed to be very discouraging, but inquiry elicited the fact that some manufacturers of china were already grinding the whole batch. This simplified the problem immensely, and a second series of experiments was undertaken. Batches of 1000 grams each were then ground with 3.5 grams sodium silicate, 3.5 grams sodium carbonate and 0.2 grams cobalt sulphate, together with a sufficient quantity of distilled water to produce a casting slip of the correct weight, *viz.*: 155 grams to 100 cc.

Body A, English china clay MGR 1. 1000 grams dry mix required 410 cc. distilled water to produce the proper slip.

Body B proved so unsatisfactory that it was not repeated.

Body C, Peach kaolin. 1000 grams required 460 cc. of water.

Body D, Newark kaolin. 1000 grams required 550 cc. of water.

Body E, Dillsboro, N. C. kaolin. 1000 grams required 560 cc. water.

Body F was not repeated as the mixture could not be vitrified.

Body G, Georgia clay. 1000 grams required 660 cc. of water.

Body H, Florida clay. 1000 grams required 740 cc. of water.

Other bodies were then made, using more than one kaolin, the total being the same as when the single kaolins were used.

Body N, equal parts of Newark and Dillsboro clays. 1000 grams required 775 cc. of water.

Body O, equal parts of Florida clay and Dillsboro kaolin. 1000 grams required 675 cc. of water.

Body P, equal parts of Georgia clay and Dillsboro kaolin. 1000 grams required 600 cc. of water.

The cast cups were burned in two batches in a commercial kiln, one burn at cone 9 and the other at cone 10. All were then glazed with a regular china glaze.

Body A made from English china clay MGR 1 was used as the criterion.

Bodies C, D, G, N, P, were all more or less dirty in translucency, though there was not much to choose between them in color when viewed by reflected light.

Bodies A, E, H, O, were a good white and clear in translucency. For all purposes body O proved the best, as H did not cast well. Body E, made from N. C. kaolin is quite equal to A in appearance, and the addition of Florida clay as in O rather improves the plasticity of the body without destroying the color. Shrinkage measurements were not made, but it may be noted that all the bodies from domestic materials required more water to produce a casting slip than that required by the body made from English kaolin. From these experiments it would appear, that from exclusively domestic materials, it is possible to produce chinas, which in appearance are quite equal to those manufactured from imported kaolin.

It will be noted from an inspection of the samples that there is a marked difference in color between the bodies burned at cone 9 and those burned at cone 10. This difference is more marked in reflected than in transmitted light. This fact seems to show that cone 10 is about the minimum temperature at which a successful china can be made.

CRAZING TEST FOR WALL TILES

BY FORREST K. PENCE

The problem of glaze fit is always a vital one to those who engage in the manufacture of glazed ceramic product. Any method whereby assurance may be obtained at any time as to the satisfactory relation between glaze and body is of distinct service.

Of course this quality of glaze fit may be ascertained by subjecting the ware to the conditions of its commercial use and noting the results over a corresponding period of time. But such delay is unsatisfactory since the information is frequently desired previous to some change in formula that may be necessary or in detecting effect of unexpected variation in raw materials.

There has also developed in the field of competition, the practice on the part of some manufacturers of guaranteeing their product, particularly white bright glazed wall tiles, against the development of crazing. Such a guarantee usually provides that if crazing develops within one year, the manufacturer shall replace the tiles with others of satisfactory quality and shall also bear the incidental expenses. Such a provision is open to abuse by reason of the abnormal or unreasonable conditions that may obtain in the placing of the tiles. The tiles may be made to fail through undue strain, defective setting, etc.

Under this guarantee, claims may also be presented which the manufacturer feels obliged to settle rather than hazard the expense of an investigation and the possibility of offending a customer.

On the other hand it would certainly be more satisfactory to the customer as well as to the final owner, to be able to have the tiles accepted or rejected on their merits, before they should be placed in the job. Therefore a standard test, that may be

applied in a short time and that will show whether or not the tiles will craze when placed in a wall under normal conditions, would be a far more satisfactory basis for a guarantee than that of the present practice.

Various methods have been used and proposed for determining the tendency of glazed ware toward crazing. Among these are the boiling water to ice water test,[1] the crystallization action of certain salts, as common salt,[2] sodium thiosulphate,[3] etc., the heating of ware to definite temperature and quenching in water,[4] etc. The application of some of these tests, especially the last, to wall tiles is known to be practiced.

Since tiles are brought so intimately into contact with the cement mortar in which they are set, we would expect the action of the cement to be an important factor in addition to the influence of temperature change. We have noted tiles slabbed in cement to craze within a few days, while free tiles from the same lot were not crazed after a number of months, although the temperature treatment of all the tiles had been the same.

As a working basis for this investigation, tiles 6 in. by 3 in. straights and 3 in. by 3 in. combination angles which were sold as first quality product of the representative tile manufacturers of this country were secured. Of the tiles, from each manufacturer, 15 straights and 6 combination angles were placed in a slab of cement mixture, one part of sand to one of Portland cement. The slabs were 1½ in. thick and approximately square. Cull tiles were set in the back of the slab to equalize the strain developed in the setting.

The remainder from each lot of tiles were held for use in making the rapid tests, slabs and free tiles being kept in a room where temperature changes were slight. Before setting forth the results of these various tests, the action of the tiles in the cement slabs will be given.

In determining the presence or absence of crazing in a tile it is sometimes necessary to apply a thin coating of colored fluid, as ink, when upon wiping the piece the color will remain in the

[1] Stover, *Trans. Amer. Cer. Soc.*, Vol. XI, p. 89.
[2] Ashley, *Trans. Amer. Cer. Soc.*, Vol. IX, p. 61.
[3] Cobb, *Trans. Amer. Cer. Soc.*, Vol. XI, p. 65.
[4] Harkort, *Trans. Amer. Cer. Soc.*, Vol. XV, p. 368.

craze mark. Marks that are only scratches show no color and are thus not taken into account.

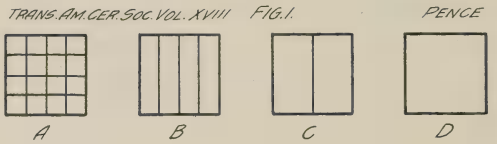
The observations on straights and combination angles are given separately as a difference in the pressing of these tiles usually affects the results.

Observations given were made 14 months after tiles were placed in the slab.

The results are shown in the Fig. 2, in which the letters A, B, C, etc. indicate the various makes of tiles and the squares opposite a given letter representing the individual tiles of that make and showing their action in the slab.

Crazing 'badly', in which distance between craze marks averages approximately one quarter inch or less, is indicated by Fig. 1a.

Crazing 'moderately', in which distance between craze marks ranges between half inch and inch, is indicated by Fig. 1b.



Crazing 'slightly', in which in some instances only one mark was found, is indicated by Fig. 1c.

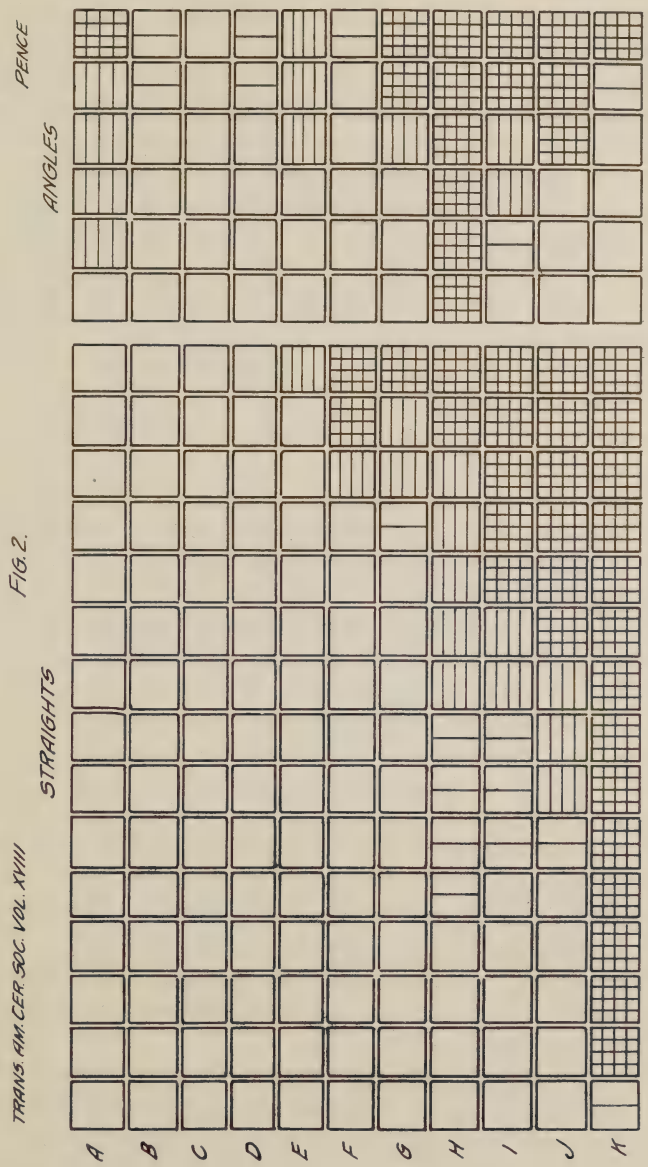
'No crazing' is indicated by Fig. 1d.

Thus of tiles 'J', six straights crazed badly, three moderately, one slightly, and five stood.

For convenience in making comparisons, the various makes of tile have been arranged and lettered in the order of the tendency of the straight tiles to craze in the slab.

In general, the straights were used in applying the various rapid tests. Where angles were used, they will be so designated. Since fewer angles were available for testing than straights, and because greater variation may also be expected in the former, the straights are the chief basis of the investigation.

For the rapid tests tiles were taken at random from the various lots and results noted in comparison with those of Fig. 2.


TRANS. AM. CER. SOC. VOL. XVIII
FIG. 2.
STRAIGHTS
ANGLES
PENCE
A
B
C
D
E
F
G
H
I
J
K

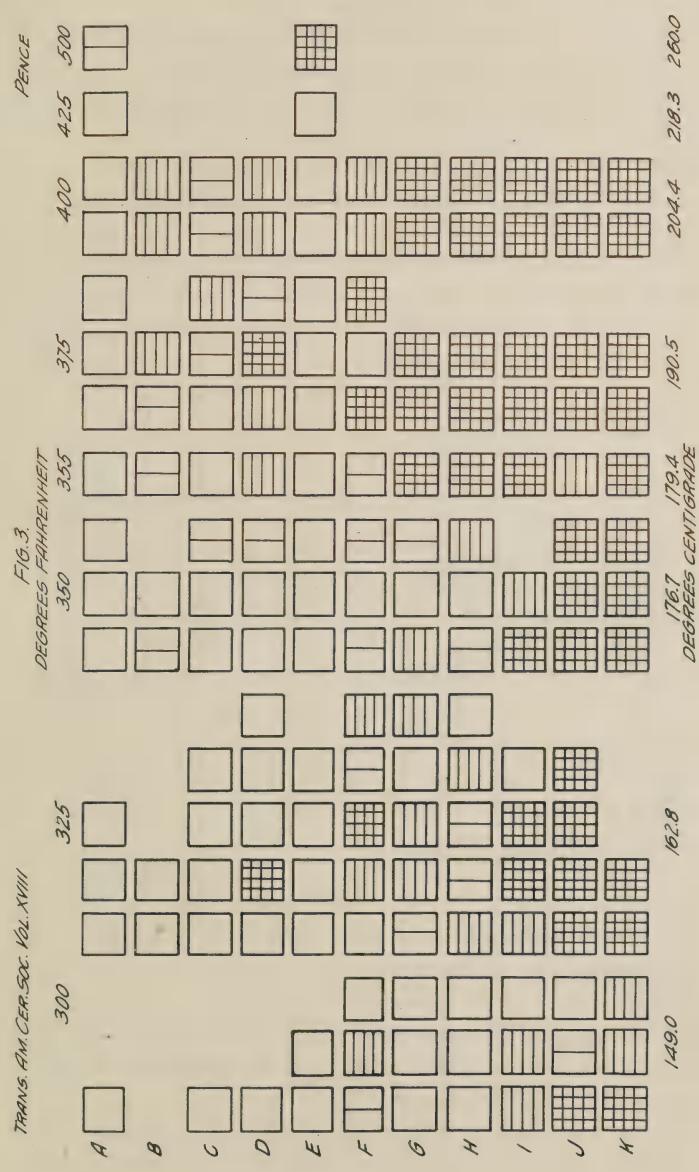
Crystallization tests were applied beginning with commercial sodium chloride (common salt).

The tiles were soaked 24 hours in concentrated solution of common salt, and dried at about 30° C. No crazing developed. The same tiles were then boiled one-half hour in the concentrated salt solution and dried at 105° C. This process was repeated four times with no development of crazing. Finally the eight hour boiling test as described by Ashley was applied, only in this case the glaze was not chipped as the porosity of the tile body permitted it to be freely permeated without this. The chipping process is also objectionable in that it could scarcely be possible to standardize it. Observation of the tiles after eight hours of boiling showed that 'J' was crazed moderately. After drying at 105° C., 'J' was crazed badly, 'I' and 'K' were crazed moderately. No other crazing developed.

While this test develops some crazing, it does not appear as severe as the cement treatment. The crazing is comparatively slight, considering the number of treatments to which the tiles were subjected.

Another crystallization test was applied in the use of sodium thiosulphate, which has been similarly used by Cobb. When gentle heat is applied to this salt it fuses or melts in its own water of crystallization. Tiles soaked a few minutes in this hot solution until solution had permeated the body as shown by darkening of entire face were allowed to cool at 5° C. The result in this case was that not only were all tiles severely crazed, but the body was ruptured, developing numerous cracks and a twisting or warping of the tile. Tiles that were soaked a very short time so that solution darkened the face for distance of one quarter inch around edge, and cooled at 5° C., developed lines of craze marks close together in the area corresponding to the distance solution had penetrated. This test is therefore much too severe.

In this connection the results of a freezing test are of interest. Tiles from all lots except 'B' and 'C', the supply of these last having been exhausted, were boiled in water ½ hour and allowed to soak in same water 14 hours. These were then allowed to freeze at 10° C. with result that 'D', 'E', and 'F', were crazed


TRANS. AM. CER. SOC. VOL. XVIII
FIG. 3
DEGREES FAHRENHEIT
DEGREES CENTIGRADE
PENCE
300
325
350
355
375
400
425
500
600
149.0
162.8
176.7
179.4
190.5
204.4
218.3
260.0
A
B
C
D
E
F
G
H
I
J
K

slightly; 'G' was crazed moderately, and 'A', 'H', 'I', 'J', and 'K' crazed badly. The result parallels that of the cement slab fairly well, with general evidence of increased severity. The result of freezing was in no case so severe as that obtained in the thiosulphate test.

The above crystallization tests do not appear entirely satisfactory as offering a rapid method of paralleling the results obtained in the cement slab.

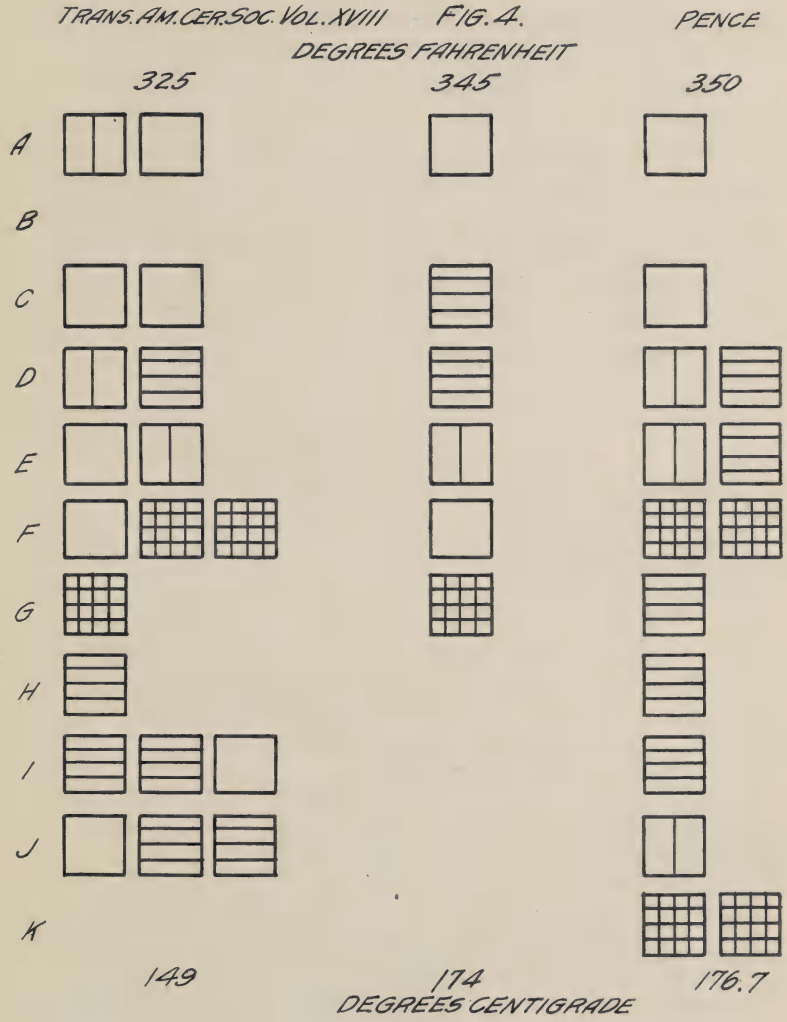
On the basis of difference in coefficient of expansion of body and glaze as a cause of crazing, a method whereby the tile should be subjected to a rapid change in temperature commends itself.

As a first step in the application of a series of such tests, the boiling water to ice water test was used. The tiles were boiled fifteen minutes and transferred quickly to water containing a liberal quantity of ice. At first treatment, the only crazing developed was one mark on tile 'J'. Upon first repetition, crazing of 'J' increased to two marks, and one mark developed on 'K'. Repetition to five treatments developed moderate crazing in 'J' and slight crazing in 'K'. No other crazing occurred. This test is therefore not sufficiently severe.

A series of quenching tests were now made in which the tiles were heated to a definite temperature and transferred quickly to ice water at the freezing point. The ice water is so defined as it was found that unless a liberal quantity of ice was used, the temperature of the water might be several degrees too high. Ice water is used to facilitate the obtaining of a standard temperature for the quenching.

The tiles were heated in a brick muffle kiln of outside dimensions 6 ft. by 6 ft. by 8 ft. The chamber was kept closed except for a removable brick for introduction of tiles. Tiles were handled by iron rod that did not come in contact with the face. The tiles being supported in the kiln by tracks at either end of the piece. It was found necessary to allow the tiles to remain at the desired temperature for one-half hour in order to insure their thorough heating.

The results of the quenching tests applied to the straights are shown in Fig. 3. Same for the angles are shown in Fig. 4. Symbols are same as in Fig. 2.


TRANS. AM. CER. SOC. VOL. XVIII FIG. 4.
PENCE
DEGREES FAHRENHEIT
325
345
350
A
B
C
D
E
F
G
H
I
J
K
149
174
176.7
DEGREES CENTIGRADE

We find in the quenching tests a development of crazing that corresponds very closely to that found in the slabs. The variations noted are such as would be accounted for by the variations in the tiles themselves, and are like those noted in tile slabs.

In the adoption of a standard test, it remains to decide to what temperature the tiles should be heated before quenching in ice water.

From a comparative study of the charts, we believe that heating to 163° C., offers a fair test. The temperature interval thus afforded corresponds to the one that Harkort found to denote a quality of safety in applying a similar test.

We therefore propose the quenching test of 163° C. (325° F.) to ice water at 0° to 1° C. (32° to 34° F.) as a reasonable substitute for a guarantee against crazing of wall tile in a normal setting.

A STUDY OF BRISTOL GLAZES COMPOUNDED ON THE EUTECTIC BASIS

BY ARTHUR S. WATTS.

The study of Bristol glaze compounds calculated on the norm basis was not thoroughly satisfactory, because of the recognized fact that these so-called norms are not the most fusible silicate or aluminium silicate of that base. If the development of the physical properties of a glaze is due to the separation of certain minerals from the magma or the development of those minerals in that magma, then the lower the temperature at which we can develop a molten magma containing these minerals or their constituents, the lower will be the temperature at which this type of glaze may be utilized.

The study by H. H. Hanna, Vol. XVII, *Transactions of the American Ceramic Society,* page 672, was conducted for the purpose of determining the most fusible mixture of K_2O-CaO-Al_2O_3-SiO_2 in which the K_2O was introduced in feldspar, the CaO in whiting, the Al_2O_3 in feldspar and kaolin, and the SiO_2 in feldspar, kaolin and free silica. As preliminary studies, he sought the most fusible mixture of Canadian feldspar and Florida kaolin and apparently found a eutectic of 92 percent feldspar, 8 percent kaolin. He next investigated the most fusible mixture of Canadian feldspar and flint and found an eutectic of 96 percent feldspar, 4 percent flint. He next sought the most fusible CaO-Al_2O_3-SiO_2 mixture, consisting of whiting, kaolin, and quartz. This he found to be CaO-.348 Al_2O_3-2.489 SiO_2. Having found these three most fusible mixtures, a series was laid out on the three component plan having as its corner members, the feldspar-quartz eutectic, the feldspar-kaolin eutectic, and the CaO-Al_2O_3-SiO_2 eutectic. A study of the fusion behavior of this series indicated that the most fusible mixture consisted of 60 percent feldspar-quartz eutectic, 40 per-

cent CaO-Al_2O_3-SiO_2 eutectic. Introduction of the feldspar-kaolin eutectic tended always to harden the mixture. The most fusible mixture produced has a formula:

$$\left.\begin{matrix}.6\ KNaO \\ .4\ CaO\end{matrix}\right\}\ .74\ Al_2O_3\text{-}4.83\ SiO_2$$

Cones of this composition deform with cone 3 or at approximately 1190 degrees C.

This is more easily fusible than the mixture reported by Seger, Vol. I, pages 224-249, *Collected Writings of Seger;* but in all these studies the use of ZnO as a flux is not considered, and it seemed probable that a fruitful field of research lay in the study of K_2O-CaO-ZnO-Al_2O_3-SiO_2 mixtures from the eutectic basis.

In the norm study the ZnO was introduced as artificial willemite, but for this study the eutectic mixture of ZnO-Al_2O_3-SiO_2 is necessary.

As it is important that the Al_2O_3 throughout this study be introduced from kaolin or feldspar, the range of compositions studied was limited to those in which the ratio of Al_2O_3 to SiO_2 was equal to or less than 1 to 2. The excess SiO_2, where present, was introduced as ground flint.

ZnO—Al_2O_3—SiO_2 STUDY

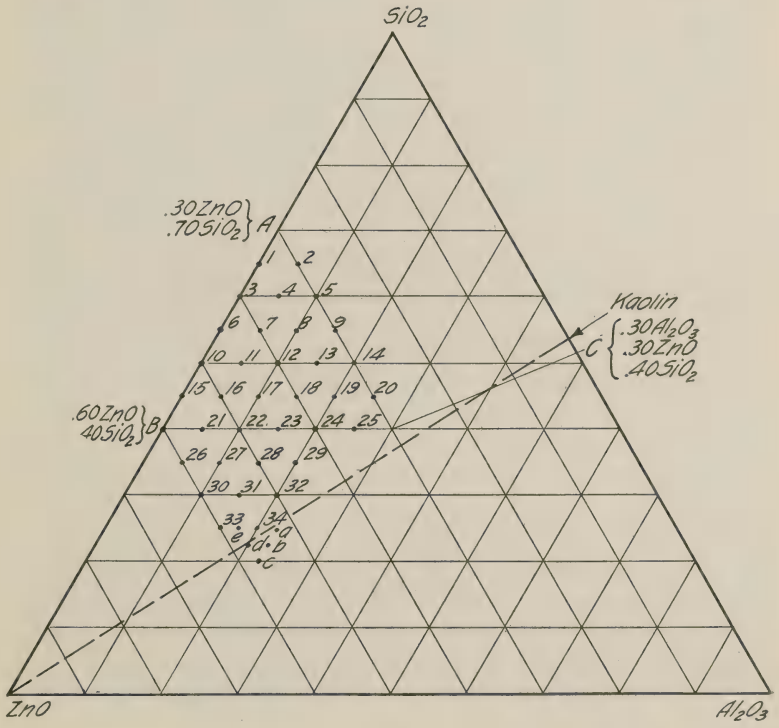
Series A. This study was divided into three parts. The first or Series A consisted of A, B, and C made up as slips, dried and then blended in powder form to produce members 1 to 25, inclusive, which were molded into cones for testing deformation. Of this series the most fusible member was found to be No. 23, which began to deform as cone 13 started, but which deformed very rapidly. Member 22 is only a few degrees harder than No. 23, and both have fused from the points down, forming globules before cone 13 is half down. As Members 23 and 22 were on the border of the series, an additional series was necessary. See Fig. 1.

Series B. Series B was prepared, consisting of members 26 to 34, inclusive. The additional members were each ground

separately, since the third member of this triaxial could not be produced without using some other alumina than that from kaolin.

The firing of Series A and B together showed Member 32 to be first to deform. It begins to deform when cone 12 just touches the plate, and deforms very rapidly so that it is down

TRANS. AM. CER. SOC. VOL. XVIII FIG. 1. WATTS



before cone 13 is one-fourth down. Member 28 is the next to deform, and follows Member 32 very closely. It in turn is closely followed by Members 22 and 23, and these check the first burn by being down when cone 13 is hardly half deformed. A fact worthy of note is that Members 32 and 28 deform without any evidence of high surface tension, while Members 22 and

23 repeat their performance of the first burn by fusing into globules at the point and gradually increasing the globule as the cones fused. This tendency to bead on deforming would mislead any one studying the fused cones if they had not data on the actual order of deformation.

To check the location of Member 32 as the most fusible ZnO-Al_2O_3-SiO_2 mixture, another burn was made of all members showing evidence of fusibility, and to these were added 32a, 32b, 32c, 32d, and 32e. This burn also included Members 51 to 55, inclusive. The result of this burn established the following order of fusibility, all within the range of cones 12 and 13:

Member 32 begins and completes deformation first. Member 32e begins and completes deformation second. Members 28 and 29 begin deformation third, but deform slowly and are not completely deformed until after Member 23. Member 22 also completes deformation slightly ahead of Members 28 and 29. Member 31 begins to deform notably behind the other members named, and fusion progresses very slowly. The tenary deformation eutectic therefore appears to be:

Series B, Member 32, composition 50 percent ZnO, 20 percent Al_2O_3, 30 percent SiO_2.

Formula: ZnO-.318 Al_2O_3-.81 SiO_2.

Temperature of deformation — cone 12-13, approximately 1380 deg. C. The physical behavior of this series of cones is shown in Fig. 2 (top).

Having determined the deformation eutectic of ZnO-Al_2O_3-SiO_2 and having previously obtained the deformation eutectics of CaO-Al_2O_3-SiO_2 and of feldspar-quartz, we are prepared to study the eutectic of these eutectics.

This study was outlined as indicated on Figure 3, the corner members being:

D A — $KNaO$, Al_2O_3, 6.4 SiO_2 — molecular wt. 580, combining wt. 580.

D B — ZnO, .318 Al_2O_3, .81 SiO_2 — molecular wt. 162, combining wt. 173.4.

D C — CaO, .348 Al_2O_3, 2.49 SiO_2 — molecular wt. 241, combining wt. 284.

The preliminary study was indicated as Series D and was produced by wet grinding the corner members and then blending to form the intermediate members D 1 to D 18, inclusive,

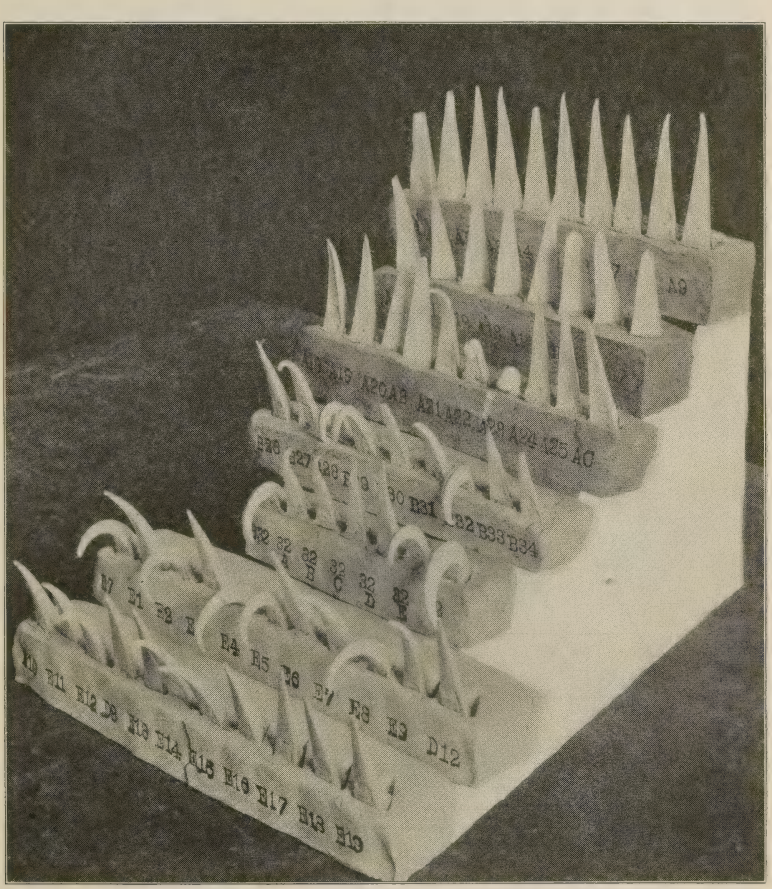
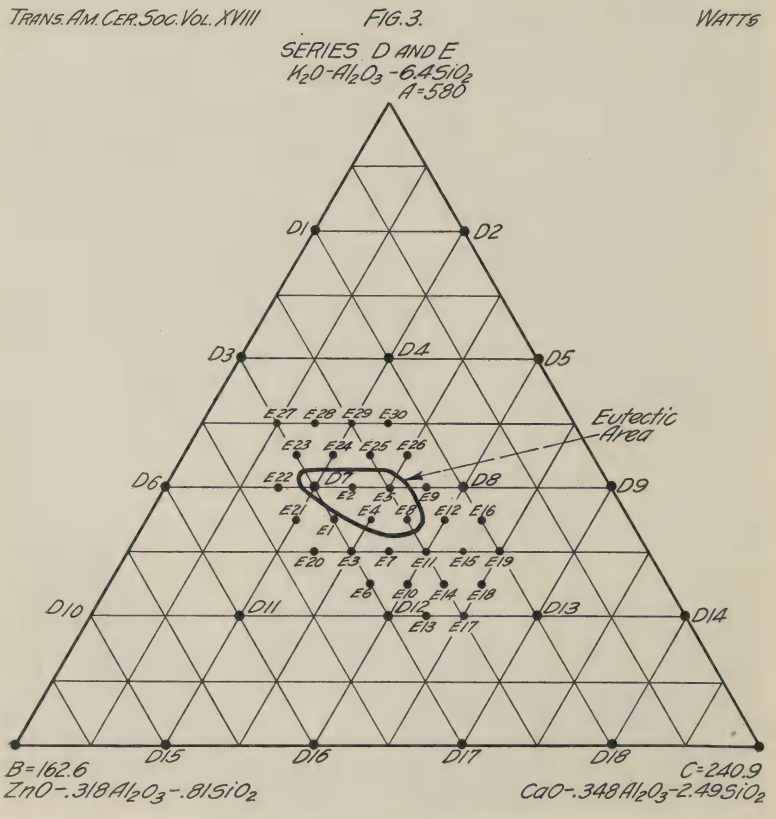


Fig. 2.

which were molded into cones for deformation determination. Of this series, D 7 deformed first and at cone 02. It was followed at about cone 01 by D 12, which was followed between cone 01 and cone 1 by D 8 and D 4 in the order named.

It therefore appeared that the most fusible mixture approached the composition of D 7 and probably was located in the area bounded by D 7, D 12 and D 8.

A new series was therefore constructed with these three members as the corners and was designated as Series E. This



series originally consisted of 15 members, viz., D 7, D 8, D 12 and E 1 to E 12, inclusive. After a preliminary burn the series was extended to E 30, inclusive (see Fig. 3).

The members were made up in the usual manner and formed into cones. These cones were tested, and an eutectic area was found consisting of mixtures D 7, E 2, E 4, E 5 and E 8. These

mixtures all deform at so near the same temperature and rate that the actual eutectic mixture was only determined to our satisfaction after repeated tests. All these mixtures deform within less than one-half cone temperature range. The temperature is cone 03 from one-fourth to three-fourths deformed.

The eutectic mixture was found to be D 5, followed in order by E 8, E 4, E 2 and E 7. The rate of deformation increases slightly toward E 8. The fusible members of this series are shown in Fig. 2 (bottom).

The most fusible mixture of modern Bristol glaze type as determined by study on the basis of eutectics is:

$$\left.\begin{matrix} .4\ KNaO \\ .3\ CaO \\ .3\ ZnO \end{matrix}\right\} .60\ Al_2O_3 \quad 3.55\ SiO_2$$

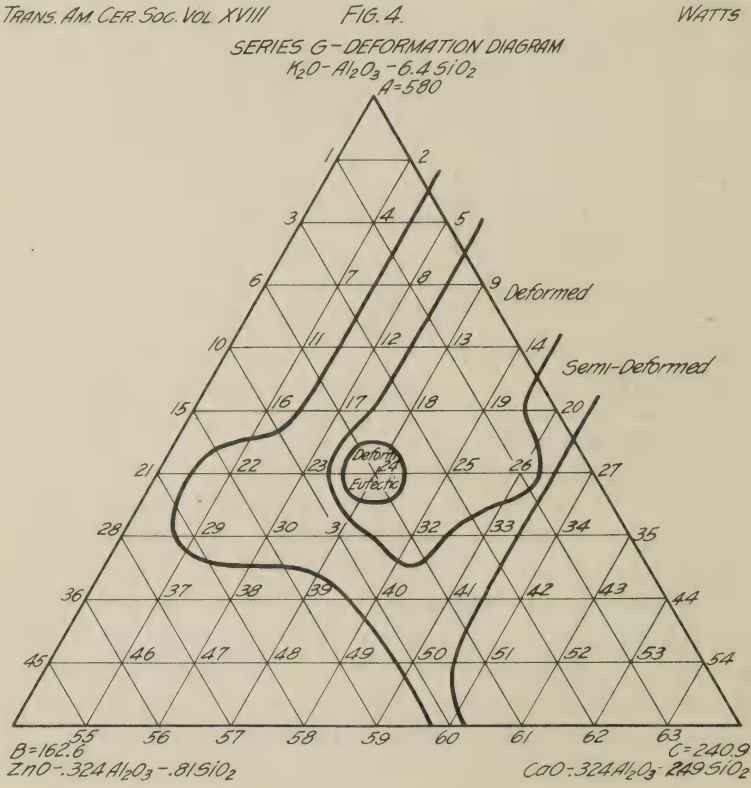
Deformation temperature, Cone 03

	percent
Canadian feldspar	59.62
Whiting	8.03
Zinc oxide	6.50
Florida kaolin	13.81
Flint	12.04
	100.00

To confirm the conclusions reached, the most fusible area was covered by an entirely new series of mixtures, and the deformation temperatures determined. The results of this test showed that while the deformation eutectic remained constant, the order and rate of deformation was somewhat affected by the rate of heat application. The slower the rise in temperature, the less active is the ZnO member.

As a final study, an entirely new series was prepared embracing the entire triaxial and designated as Series G. (See Fig. 4) The eutectics were newly prepared, and after blending wet, the mixtures were thoroughly ground in a mortar. Stoneware trials were dipped in each mixture, and the remainder was dried to leather hardness and molded into cones.

Series G consists of the three eutectic mixtures and sixty-three intermediate mixtures blended on the molecular basis. This gives a molecular variation of .1. The cones were tested for deformation order, and the preceding observations again confirmed. The variation of mixture over the entire triaxial gave a far clearer study of deformation behavior, and the temperature



was increased until evidence of deformation was apparent along two margins of the series. The range of composition within which deformation occurs at cone 4 is shown by the accompanying illustration, Fig. 5.

The deformation eutectic is clearly G 24, with G 25 only slightly more refractory. Toward the ZnO extreme of the series,

the cones show evidence of interior fusion, while their surfaces appear dry. This would point to volatilization. Toward the

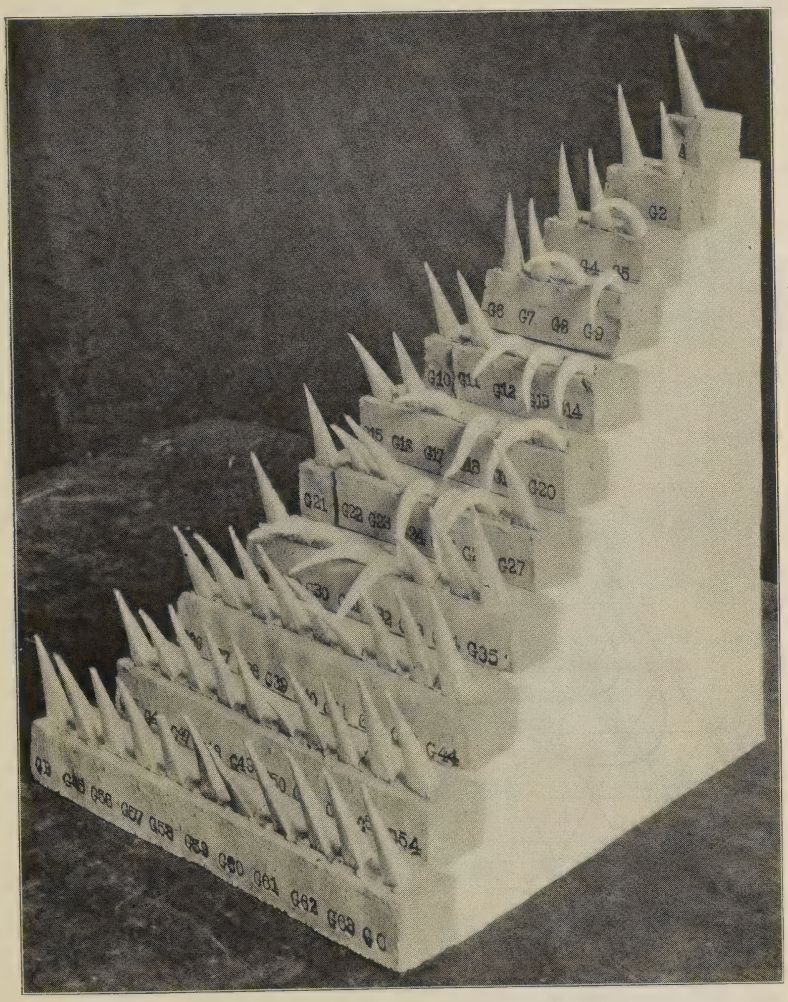
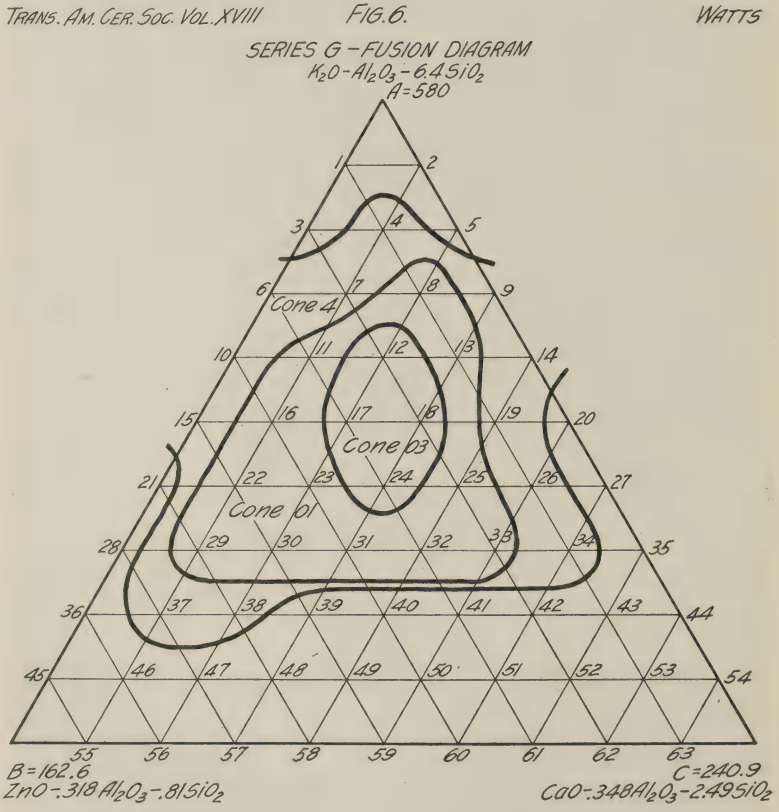


FIG. 5.

CaO extreme of the series the rate of deformation is more gradual, the vitrifying action being apparent in all the cones in this corner of the series. Toward the K_2O extreme of the series,

the development of a glossy structure is very apparent although the deformation did not begin until the members lower in feldspar had progressed far in the deformation process. One would, therefore, naturally assume that glassy glazes develop on the K_2O side of the eutectic. A study of the stoneware trials which



were glazed with the various mixtures of this series will show this assumption to be correct.

GLAZE TRIAL STUDY

The stoneware trials glazed with members of this series were fired at cones 03-01 and 4. (Fig. 6).

The progress of glaze development is indicated on Fig. 6, and it will be noted that this does not exactly coincide with order of deformation as evidenced by the cone study.

In general it may be said that with G 24 as the eutectic both for deformation and glaze development, there is a rapid increase of glassiness with K_2O increase, a rapid increase of white opacity with ZnO increase and a distinct increase of cream opacity with CaO increase.

In conclusion, it should be pointed out that a change in composition due to ZnO volatilization may cause a slight variation in the development of both cones and glazes toward the ZnO extreme of this study, but duplication of tests has failed to develop any evidence of other possible variations.

A COMPARISON BETWEEN SODIUM SULPHATE AND FREEZING TESTS FOR DRAIN TILE

BY HOMER F. STALEY.

PART I. FREEZING TESTS

The freezing tests here described were made by W. J. Schlick, Drainage Engineer, and R. W. Crum, Structural Engineer, of the Iowa State College Engineering Experiment Station.

The Tile Tested. The tests were made upon hard and soft 8 in., 12 in., and 16 in. clay drain tile from four representative Iowa factories and upon average good 8 in. and 16 in. concrete tile from one representative Iowa factory. In selecting the clay tile the hardest burned, but not overburned, and the softest burned tile from one kiln were selected whenever possible.

The concrete tile were shipped from the factory as being representative of their average good product. In all cases, tile of a clear ring and free from cracks were selected. The hard clay tile were selected in each case as being representative of the best of the No. 1 product of that factory. In each case the pipe were donated for these tests by the factories. It is questionable whether the concrete tile used in these tests are fairly representative of the best quality of concrete tile, and another series of tests on a wider variety of concrete tile is now under way.

The list of factories from which tile were procured and a short description of the tile selected follows:

Factory L. The concrete tile used were shipped from the above factory, by the plant management. The president of the above company describes the pipe in a letter as follows:

"These tile are sixty days old, as near as we can tell, are made of washed sand and Northwestern cement, proportions three to one, and steam cured for forty-eight hours, after which time they were rolled out into the yard."

Factory M. The 16 in. tile from this plant were selected from a portion of the stock pile which the yard men stated were made up of the tile from the last kiln of 16 in. tile fired. It is thought that all of the 16 in. tile are from one kiln and represent the extremes of the product from that kiln. All might have been classed No. 1, though the soft tile might be excluded under a severe inspection.

The 8 in. tile were selected from the stock pile and represent the hard and soft tile of this stock pile.

Factory N. The 16 in. tile from this factory were chosen as the tile were removed from the kiln and would probably all be classed as No. 1.

The 8 in. tile were selected from the stock pile. The soft tile were hunted out and are somewhat softer than the average soft tile of this size from this plant. The superintendent stated that the soft tile were selected from two kilns which were in course of firing at a time when he ran short of fuel oil.

Factory P. All of the tile selected, both 8 in. and 16 in., were chosen as the tile were removed from the kilns and represent the hardest and softest from these kilns. It is thought that the specimens selected are fairly representative of the extremes in the No. 1 tile from this factory.

Factory R. The tile from this factory were selected from the stock pipe and represent the hard and soft 12 in. tile in the yard at the time the selection was made.

Selection of Samples. Strength tests were made upon five samples of each size and class of tile from each factory. From each of these tested tile, six samples were obtained for absorption, freezing and thawing and sulphate tests.

Each specimen was approximately square, of from 10 to 20 sq. inches in area and of the full thickness of the pipe wall. Where the appearance of the tile indicated that it was of uniform quality throughout, the specimens were chosen from the body (not from the end), and at the sides of the segments of the tested tile. The specimens were taken from different portions of the tile so as to be representative of the whole pipe wall. After the completion of the absorption tests, two of the pieces from each tile were subjected to the accelerated freezing and

thawing tests (First Series) and the other four retained for the sulphate tests and additional freezing and thawing tests.

Five additional tile of each size and class from each factory were subjected to the natural freezing and thawing test. At the completion of this test, the tile were subjected to the standard strength tests. No notable decrease in strength was observed. Details are not given in this paper.

ACCELERATED FREEZING TESTS

First Series. These tests were made upon two specimens from each tile. All the specimens for this test were packed in the basket of the freezing box, which was kept packed with crushed ice and salt. They were subjected to temperatures of from 3° F to 7° F for at least five hours at each freezing period. They were thawed by placing in water at a temperature slightly below boiling, for one hour. For the first 50 freezings and thawings the specimens were taken from the hot water and allowed to cool in air before replacing in the freezing box. In starting the second 50 freezings and thawings this program was varied, and the specimens were cooled in water at 70° F. The specimens were given two freezings and thawings per day.

The absorptions of the pieces tested are given in Table I, and in Table II are given the completed data of the effect of this series of freezing and thawing tests. The data are complete for 100 freezings and thawings except for pieces No. 47A and 81 to 90 inclusive, which are still being tested. In stating the effects of the freezings and thawings in this table, the effort has been made to describe only those conditions which were due to the freezings. No especial examination was made of these specimens before these tests were started, but as each specimen was examined carefully after each tenth freezing, it is possible to determine the effect of the freezings very closely.

Second Series. After the completion of the first series of freezing and thawing tests, it was decided to start a second series in which the specimens would be subjected to more severe conditions.

The specimens for this series were the third or remaining specimen of the first three specimens from each tile, the other

TABLE I. ABSORPTION OF PIECES USED IN ACCELERATED FREEZING TESTS

First Series

PIECE NO.	ABSORPTION 72 HOURS IMMERSION	CLASS	PIECE NO.	ABSORPTION 72 HOURS IMMERSION	CLASS
1 B	9.23	16 in. Concrete, Factory L....	23 C	2.70	
1 C	9.97		24 A	3.50	
2 B	10.94		24 B	3.37	
2 C	10.35		25 A	4.12	
3 B	9.28		25 C	4.54	
3 C	7.56				
5 B	8.58		26 B	2.30	16 in. Clay Hard, Factory M.
5 C	9.13		26 C	2.01	
6 A	9.11		27 A	3.01	
6 C	10.17		27 B	3.05	
			28 A	2.05	
11 B	9.09	8 in. Concrete, Factory L....	28 B	2.83	
11 C	8.53		29 B	2.16	
12 A	8.71		29 C	1.90	
12 B	8.39		30 A	2.86	
13 A	7.43		30 C	2.61	
13 B	8.42				
14 A	8.29		31 A	8.78	8 in. Clay Soft, Factory M....
14 B	7.68		31 C	8.46	
15 A	8.87		32 B	7.56	
15 C	9.86		32 C	7.86	
			33 A	7.58	
21 A	4.24	16 in. Clay Soft, Factory M....	33 B	8.06	
21 B	4.72		34 A	7.26	
22 B	5.48		34 B	7.91	
22 C	4.84		35 A	6.45	
23 B	2.35		35 C	5.99	

TABLE I — Concluded

PIECE NO.	ABSORPTION 72 HOURS IMMERSION	CLASS	PIECE NO.	ABSORPTION 72 HOURS IMMERSION	CLASS
36 A	4.61	8 in. Clay Hard, Factory M...	48 A	1.93	
36 C	4.80		48 C	2.13	
37 B	4.77		49 A	3.46	
37 C	4.70		49 C	3.17	
38 B	4.76		50 B	3.55	
38 C	4.90		50 C	3.17	
39 A	6.85				
39 C	6.79		51 A	5.21	8 in. Clay Soft, Factory N....
40 A	5.91		51 B	5.84	
40 B	5.52		52 A	4.18	
			52 C	4.43	
41 A	5.54	16 in. Clay Soft, Factory N....	53 A	2.43	
41 B	4.17		53 B	2.28	
42 A	4.96		54 A	6.99	
42 B	4.30		54 B	6.58	
43 A	4.38		55 A	4.07	
43 C	4.07		55 C	4.06	
44 A	3.82				
44 B	3.96		56 A	2.89	8 in. Clay Hard, Factory N...
			56 G	2.88	
45 A	4.14		57 A	2.58	
45 C	4.33		57 C	2.77	
			58 A	2.64	
			58 C	2.46	
46 A	2.08	16 in. Clay Hard, Factory N...	59 B	2.73	
46 C	1.79		59 C	2.35	
47 A	3.96		60 A	2.63	
47 C	3.81		60 B	3.37	

61	A	9.70	16 in. Clay Soft, Factory P....	71	A	12.95	8 in. Clay Soft, Factory P....
61	C	9.32		71	B	12.96	
62	A	8.81		72	A	10.78	
62	B	10.36		72	B	11.23	
63	B	7.02		73	B	13.50	
63	C	7.64		73	C	12.86	
64	B	9.61		74	A	13.35	
64	C	8.67		74	B	13.03	
65	A	9.73		75	A	13.90	
65	C	9.95		75	C	14.30	
66	B	10.45	16 in. Clay Hard, Factory P...	76	B	9.23	8 in. Clay Hard, Factory P...
66	C	11.13		76	C	9.31	
67	A	6.85		77	A	11.80	
67	B	6.73		77	C	9.06	
68	B	8.33		78	A	10.41	
68	C	8.70		78	B	9.25	
69	B	12.94		79	B	9.80	
69	C	12.62		79	C	10.18	
70	A	8.00		80	A	8.70	
70	B	7.77		80	B	9.18	

TABLE II. RESULTS OF FREEZINGS AND THAWINGS, PIECES FROM TESTED TILE

Accelerated Tests. First Series

PIECE NO.	ABSORPTION 72 HOURS IMMERSION	50 FREEZINGS AND THAWINGS — VISIBLE EFFECT	CHANGE IN DRY WEIGHT	100 FREEZINGS AND THAWINGS — VISIBLE EFFECT	CHANGE IN DRY WEIGHT
		16 in. Concrete, Factory L			
	percent		*percent*		*percent*
1 B	9.23	O. K.	—0.13	Destroyed, can be broken with hands	.....
C	9.97	Crumbling badly at edges	—8.62	Destroyed, can be broken with hands	.....
2 B	10.94	Crumbling slightly	—0.28	Destroyed, entirely broken down	.....
C	10.35	Crumbling at one corner	—1.43	Destroyed, can be broken with hands	.....
3 B	9.28	Crumbling slightly, one corner	—0.24	Continues crumbling at corner	—0.22
C	7.56	O. K.	—0.18	Crumbling slightly, one corner	—0.63
5 B	8.58	O. K.	—0.45	Destroyed, entirely broken down	.....
C	9.13	O. K.	—0.16	Destroyed, entirely broken down	.....
6 A	9.11	Crumbling slightly, one corner	—0.64	Destroyed, can be broken with hands	.....
C	10.17	Crumbling slightly, one corner	—0.64	Destroyed, entirely broken down	.....
		8 in. Concrete, Factory L			
11 B	9.09	O. K.	—0.08	Destroyed, entirely broken down	.....
C	8.53	O. K.	—0.17	Destroyed, can be crumbled with hands	.....
12 A	8.71	Crumbling a little	—0.95	Crumbling, one side	—4.56
B	8.39	Crumbling badly	—0.66	Crumbling badly	—19.63
13 A	7.43	O. K.	—0.80	O. K.	—0.82
B	8.42	O. K.	—0.17	Destroyed, can be crumbled with hands	.....
14 A	8.29	O. K.	—0.18	Destroyed, can be crumbled with hands	.....

	B	7.68	O. K.	—0.06	Destroyed, entirely broken down	
15	A	8.87	O. K.	—0.31	Destroyed, entirely broken down	
	C	9.86	Crumbling a little	—0.45	Destroyed, entirely broken down	
			8 in. Clay Soft, Factory M			
33	B	8.06	O. K.	—0.06	Crack in top	—0.15
			16 in. Clay Soft, Factory N			
43	C	4.07	Piece off at corner	—3.63	Small laminations opening	—3.79
			8 in. Clay Soft, Factory N			
51	B	5.84	Laminations opening at 3 corners	—0.09	Opened slightly. Corners crumbled slightly	—0.89
			16 in. Clay Hard, Factory P			
66	B	10.45	Many small laminations opening	—0.66	Laminations show badly. Slight scaling at edges	—1.18
	C	11.13	Piece loosened at corner	—0.17	Laminations showing badly, crumbling at one side	—1.00

TABLE II — Concluded

PIECE NO.	ABSORPTION 72 HOURS IMMERSION	50 FREEZINGS AND THAWINGS — VISIBLE EFFECT	CHANGE IN DRY WEIGHT	100 FREEZINGS AND THAWINGS — VISIBLE EFFECT	CHANGE IN DRY WEIGHT
		8 in. Clay Soft, Factory P			
	percent		*percent*		*percent*
73 B	13.50	Small lamination opening at corner.	—0.07	Many small laminations opening. Crumbling at corners. Piece off at corner..	—10.95
74 A	13.35	Small laminations opening at corner	—0.18	Destroyed, can be broken with hands.....	.....
75 C	14.30	O. K.	—2.83	Crumbling at corner. Many small laminations opening	—4.19
		8 in. Clay Hard, Factory P			
78 A	10.41	O. K.	—0.05	Crumbling at one edge; laminations openings at that edge.....	—0.46
79 C	10.18	Small laminations opening at side..	—0.05	Also piece off at corner.....	—0.81
80 A	8.70	Small laminations opening at side..	—1.87	Also piece off at corner.....	—4.47

two specimens from each tile being used for the first series of tests. In general, each specimen in this series had an absorption higher than one and lower than the other of the other two specimens, from the same tile, used in the first series of tests.

In these tests the specimens are all laid with the curved or convex side downward in trays or pans fitted in a frame which replaces the wire basket used in the freezing box in the first series of tests. The specimens were so sorted that all the samples in each tray were from tile of the same diameter and wall thickness. Each tray was filled with water to such a depth that one-third of each specimen was immersed. This one-third depth was taken as one-third the sum of the thickness of the piece plus the average concavity of the samples.

The specimens for this test had been immersed in water at room temperature for about 60 days previous to preparing for this test. They were then immersed in water near boiling temperature for three hours and then allowed to cool in water.

It was found that only one freezing and thawing per day could be completed, as the water in the trays could not be successfully frozen in the five hour period available during the day. The program was accordingly varied from that used in the first series to the following. The specimens are frozen for at least 18 hours at a temperature starting at not more than 10° F. and decreasing to, or nearly to, 0° F. at the end of the freezing period. The crate holding the trays is then placed in the heating tank, and the specimens kept in water near, or at a boiling temperature for three hours. The crate is then lifted, and the hot water poured from the pans after which it is immersed in water at room temperature for one hour. At the completion of this one hour cooling, the crate is raised, and the water poured from the trays. Each tray is then filled to the required depth with ice water, and the crate returned to the freezing box for another freezing period.

Each specimen was examined carefully before starting the freezings, and a record made of all cracks and laminations visible to the naked eye. The specimens were watched carefully for the first signs of deterioration. Careful examinations and

records were made after the 10th freezing and thawing, after the 13th and then after every other freezing and thawing.

The absorptions of the pieces tested are given in Table III. The results of this series up to and including 20 freezings and thawings are given in Table IV. In each case the cracks, openings, laminations, and other evidences of the effects of the tests given are due to the tests and were not visible when this series was begun. These tests are still being continued. Up to 75 treatments, no clay tile with less than seven percent absorption has been noticeably affected.

TABLE III. ABSORPTION OF PIECES USED IN ACCELERATED FREEZING TESTS

Second Series

PIECE NO.	ABSORPTION 72 HOURS IMMERSION	CLASS	PIECE NO.	ABSORPTION 72 HOURS IMMERSION	CLASS
1 A	9.74	16 in. Concrete, Factory L....	31 B	8.48	8 in. Clay Soft, Factory M....
2 A	10.82		32 A	7.55	
3 A-1	.....		33 C	7.59	
3 A-2	7.98		34 C	7.49	
5 A	9.05		35 B	6.83	
6 B	9.30				
			36 B	4.52	8 in. Clay Hard, Factory M...
11 A	8.96	8 in. Concrete, Factory L....	37 A	4.60	
12 C	8.50		38 A	4.71	
13 C	8.64		39 B	6.76	
14 C	7.81		40 C	5.35	
15 B	8.98				
			41 C	4.20	16 in. Clay Soft, Factory N....
21 C	4.16	16 in. Clay Soft, Factory M....	42 C	4.64	
22 A	5 .18		43 B	4.16	
23 A	2.34		44 C	3.95	
24 B	3.37		45 B	4.06	
25 C	4.54				
			46 B	2.05	16 in. Clay Hard, Factory N...
26 A	2.09	16 in. Clay Hard, Factory M...	47 B	3.88	
27 C	2.70		48 B	1.92	
28 C	2.81		49 B	3.19	
29 A	1.73		50 A	3.34	
30 B	2.88				

TABLE III — Concluded

Second Series

PIECE NO.	ABSORPTION 72 HOURS IMMERSION	CLASS	PIECE NO.	ABSORPTION 72 HOURS IMMERSION	CLASS
51 C	5.36	8 in. Clay Soft, Factory N...	71 C	12.94	8 in. Clay Soft, Factory P...
52 B	4.22		72 C	11.02	
53 C	2.10		73 A	13.10	
54 C	6.67		74 C	13.13	
55 A	3.54		75 B	13.89	
56 B	2.93	8 in. Clay Hard, Factory N...	76 A	9.62	8 in. Clay Hard, Factory P...
57 B	2.57		77 B	9.64	
58 B	2.77		78 C	9.12	
59 A	**2.73**		79 A	10.09	
60 C	2.75		80 C	8.92	
61 B	9.61	16 in. Clay Soft, Factory P...	81 B	5.54	12 in. Clay Soft, Factory R...
62 C	9.99		82 C	5.94	
63 A	7.27		83 A	5.84	
64 A	9.47		84 B	5.60	
65 B	9.72		85 C	6.23	
66 A	10.88	16 in. Clay Hard, Factory P...	86 C	4.69	12 in. Clay Hard, Factory R...
67 C	6.65		87 A	5.60	
68 A	8.55		88 B	4.69	
69 A	12.60		89 C	4.64	
70 C	7.37		90 C	5.09	

TABLE IV. RESULTS OF FREEZINGS AND THAWINGS, PIECES FROM TESTED TILE

Second Series

PIECE NO.	ABSORPTION 72 HOURS IMMERSION	FIRST NOTICEABLE EFFECT		EFFECT OF 20 FREEZINGS AND THAWINGS, UNLESS SPECIWERE DESTROYED BY A LESS NUMBER
		NUMBER OF FREEZINGS AND THAWINGS	DESCRIPTION OF EFFECT	
16 in. Concrete, Factory L				
1 A	9.74	8	Crumbling under side.....	Destroyed by 15; crumbled easily in hands.
2 A	10.82	8	Crumbling under side.....	Destroyed by 15; crumbled easily in hands.
*3 A-1	.....	15	Piece off at corner.....	Crumbling slightly one side.
3 A-2	7.98	13	Chipped at top.....	Crumbling slightly one side.
5 A	9.05	8	Crumbling, under side.....	Destroyed by 15; crumbled easily in hands.
6 B	9.30	8	Crumbling, under side.....	Destroyed by 15; crumbled easily in hands.
8 in. Concrete, Factory L				
11 A	8.96	8	Crumbling, under side.....	Destroyed by 15; crumbled easily in hands.
12 C	8.50	17	Crumbling slightly at edges.....	No further effect.
13 C	8.64	8	Crumbling at edges.....	Destroyed by 15; broken easily in hands.
14 C	7.81	15	Crumbling slightly, one edge.....	(Left out 15th to 20th by mistake.)
15 B	8.98	8	Crumbling edges	Entirely broken down by 13.

* Specimen 3 A had to be cut into two pieces (3 A-1 and 3 A-2) in order to get it in the freezing pan

TABLE IV — Concluded

PIECE NO.	ABSORPTION 72 HOURS IMMERSION	FIRST NOTICEABLE EFFECT: NUMBER OF FREEZINGS AND THAWINGS	FIRST NOTICEABLE EFFECT: DESCRIPTION OF EFFECT	EFFECT OF 20 FREEZINGS AND THAWINGS, UNLESS SPECIMENS WERE DESTROYED BY A LESS NUMBER
16 in Clay Soft, Factory P				
63 A	7.27	8	Piece off at one corner.....	No other effect.
64 A	9.47	8	Piece off at one corner.....	Another piece off at corner.
16 in. Clay Hard, Factory P				
69 A	12.60	13	Piece off at corner.....	(Left out 15th to 20th by mistake.)
....	.....	15	Scaling on bottom; structure weakening	
8 in. Clay Soft, Factory P				
71 C	12.94	8	Scaling slightly, under side.....	Destroyed by 15; can be broken in hands.
72 C	11.02	..		No visible effect.
73 A	13.10	9	Piece off at corner.....	Scaling at bottom; structure weakening.
74 C	13.13	8	Scaling at one corner, under side...	Breaking down rapidly.
75 B	13.89	8	Scaling at one corner, under side...	Destroyed by 15; split at water line.
Other samples not affected				

NATURAL FREEZING AND THAWING TESTS

Five tile of each size and class from each factory were subjected to the natural freezing and thawing tests. The tile were first thoroughly wet by immersion for 48 hours, and then allowed to freeze out of doors. Each morning they were brought inside and allowed to thaw out in tanks of water at room temperature, and each evening again placed out of doors to freeze. The tests were continued as long as the weather conditions were favorable.

Complete records were kept of the outdoor and indoor temperatures, and of the temperatures of the water in the thawing tanks. The tile in this test were out of doors on 33 nights when the temperature was freezing or below and on 8 nights when the temperature was 0° F. or below. The temperature of the water in the thawing tanks ranged from 37°F. to 62°F., and averaged near 46°F. for the days when the tile were being thawed following freezing.

Each group of pipe in this series is given a number 100 larger than the same kind of pipe in the tests of the pipe from which the samples were taken for the accelerated freezing and thawing tests. The summations of the visible results of the freezings are given in Table V.

TABLE V. EFFECT OF NATURAL FREEZINGS AND THAWINGS OF WHOLE TILE

TILE NO.	KIND	DESCRIPTION OF EFFECT
101	8 in. Concrete, Factory L.	Chipped at inside edge at top.
102		One chip from inside edge at top.
103		Chipped at inside edge at top. Several small cracks opening at the top, and some scaling inside.
104		Same as 103.
105		Same as 103.
111	16 in. Concrete, Factory L.	O. K.
112		O. K.
113		O. K. Some rough spots outside.
114		O. K. Some rough spots outside.

TABLE V — Continued

TILE NO.	KIND	DESCRIPTION OF EFFECT
115		O. K. Some rough spots outside. Chip out at top.
121	16 in. Clay Soft, Factory M.	Two chips from inside of top. Rough outside.
122		Two chips from outside at top.
123		Two cracks, 2 in. at top. Also one chip out at top.
124		One chip out at the inside and rough outside.
125		Two chips out; 1½ in. crack and two 1 in. laminations opened up.
126	16 in. Clay Hard, Factory M.	One chip out at top.
127		Two small chips from outside at top.
128		O. K.
129		Three-inch lamination opened and two chips off at top.
130		Four chips from inside at top; rough outside at top.
131	8 in. Clay Soft, Factory M.	Two chips from outside at top; 6 in. chip from top at outside.
132		Two chips from outside at top; 1½ in. lamination opened at bottom.
133		Two chips from outside at top.
134		Three chips from inside at top; edges rough.
135		Rough at top. Two small chips off at top.
136	8 in. Clay Hard, Factory M.	One chip from top and two from bottom.
137		One small chip off.
138		One chip off at top.
139		O. K. but rough at top.
140		O. K. but rough at top.
141	16 in. Clay Soft, Factory N.	Big chip off at end; also blister inside and small crack at bottom.
142		Three chips off and a lamination opening up.
143		Three chips off at each end; one small crack.
144		Two big chips off at end and three inch lamination opened up.
145		Two chips off at one end and one at the other.
146	16 in. Clay Hard, Factory N.	One chip from inside of tile.
147		O. K.
148		One chip from outside at top; two-inch blister cracking.

TABLE V — Continued

TILE NO.	KIND	DESCRIPTION OF EFFECT
149		One chip off from end.
150		O. K.
151	8 in. Clay Soft, Factory N.	One-half inch crack and three-inch lamination opening at bottom. Other small laminations showing.
152		Six-inch crack inside.
153		Seven-inch crack and one lamination opening up.
154		Four-inch crack inside.
155		One chip off at each end.
156	8 in. Clay Hard, Factory N.	Big chip from outside of top.
157		O. K.
158		O. K.
159		O. K.
160		O. K.
161	16 in. Clay Soft, Factory P.	Small laminations opening at top; one chip off and 3-inch crack.
162		Three chips off at top and one lamination opening.
163		Three laminations opening at top; one chip off at top; two-inch crack.
164		One large and one small lamination opening at the top; also blister inside.
165		Chipped at outside at top for two-thirds the circumference.
166	16 in. Clay Hard, Factory P.	Two 4-inch cracks on the inside at top.
167		Many small chips off and several small laminations open at top; also two 1-inch cracks.
168		O. K.
169		Four chips off and three laminations opening at top.
170		Three chips off at top.
171	8 in. Clay Soft, Factory P.	Small chips off and four-inch lamination opening at top; many four-inch cracks.
172		One large and one small lamination opening; two cracks half way through body; many small cracks.
173		Four chips off at top; two cracks through body at top and many cracks at the bottom.
174		One three-inch lamination opening at the top.
175		Two chips off at top; two cracks at top.
176	8 in. Clay Hard, Factory P.	Small laminations open all around top.
177		Small laminations opened at top; also two-inch crack.
178		Three-inch lamination open at the top.

TABLE V — Concluded

TILE NO.	KIND	DESCRIPTION OF EFFECT
179		Three cracks through body and two laminations opening.
180		One chip off and several laminations opening.
181	12 in. Clay Soft, Factory R.	O. K.
182		Two chips off at top.
183		Three-inch lamination opened at top; small chips from outside.
184		One chip from inside.
185		Two chips off at top.
186	12 in. Clay Hard, Factory R.	O. K.
187		One chip from inside and several small chips from outside at top.
188		O. K.
189		One large chip off at end.
190		Chipping slightly; hair crack two-thirds length of body.

PART II. SODIUM SULPHATE TESTS

Object. The object of this part of the investigation was to determine whether a sodium sulphate test can be used for drain tile in place of freezing tests. Sodium sulphate tests have been proposed, and used to a limited extent, as a substitute for freezing tests on stone since the time of Brard[1]. A modification of Brard's process has been applied to clay bricks by Howat[2] and Orton[3].

Solubility of Sodium Sulphate. The solubility of sodium sulphate in water at various temperatures is shown in Table VI. Since the solubility varies so greatly with temperature, it was decided not to use a saturated solution but one containing 15 percent of anhydrous Na_2SO_4. Such a solution is affected very little by unavoidable changes in room temperature, as long as the temperature does not drop below the saturation point of the solution 65.8°F. At its saturation temperature such a solution has a

[1] *Annales de Chemie et de Physique*, 1828, Vol. 38, page 160.
[2] *Trans. Amer. Cer. Soc.*, Vol. XVII, page 248.
[3] *Trans. Amer. Cer. Soc.*, Vol. XVIII.

density of 1.132[4]. Preliminary tests showed that ten treatments with a method (A. below) similar to that used by Howat and Orton had an appreciable effect on drain tile of the character we intended to test with the 15 percent solution. When 10 percent solutions were used, there was no noticeable effect on clay tile at the end of ten treatments.

TABLE VI. SOLUBILITY OF SODIUM SULPHATE IN WATER

TEMPERATURE° C.	GRAMS ANHYDROUS Na_2SO_4 PER 100 GRAMS SOLUTION	SOLID PHASE
0	4.76	
5	6.0	
10	8.3	
15	11.8	
20	16.3	$Na_2SO_4 H_2O$
25	21.9	
30	29.0	
31	30.6	
32	32.3	
32.75	33.6	
33	33.6	
35	33.4	
40	32.8	
50	31.8	
60	31.2	
80	30.4	Na_2SO_4
100	29.8	
120	29.5	
140	29.6	
160	30.7	
230	31.7	

NOTE: Seidell — *Solubilities,* 1911 edition, page 311.

Methods of Testing Used. Three types of tests were employed.

A. Samples were immersed in 15 percent Na_2SO_4 solution at 21°C. (70°F.) for 48 hours, then placed in a dryer at 110°C. (230° F.) for 7 hours. This constituted the first treatment. Subsequent treatments consisted of immersing the samples in the solution for 16 hours and placing them in the dryer for 7 hours. Thus, after the first, one treatment was performed each day. The

[4] Landolt, Bornstein, Meyerhoffer — *Physikalish-Chemische Tabellen,* 3rd edition, page 318.

other hour of the 24 was consumed in examining the samples, transferring them, and in allowing them to cool after they came from the dryer. In this method of drying, anhydrous Na_2SO_4 would be formed.

B. Samples were immersed in 15 percent Na_2SO_4 solution at 21°C. (70°F.) for 48 hours, then stood on edge separately on boards in a large closed room, kept at temperature between 69°F. and 73°F. for a period of 24 hours. Subsequent treatments consisted of immersion in the solution for 24 hours and drying in the room for 24 hours. In this method of drying, Na_2SO_4 $10H_2O$ and possibly Na_2SO_4 $7H_2O$ would be formed.

C. Samples were immersed continuously in 15 percent Na_2SO_4 solution. The object of this test was to determine whether the solution had any chemical effect on the samples.

Method A. The absorptions of the pieces used in this series of tests are shown in Table VII, and the results summarized in Tables VIII, IX, X, and XI. From the data in hand, it seems that one treatment with sodium sulphate by this method is approximately equivalent to two freezings by the partial-immersion accelerated freezing test. This is shown best by comparison of Tables IV and IX. This method of testing is quicker, more convenient, and more economical than any of the freezing tests, or other sulphate tests, tried. The parallelism between high porosity and easy attack by both the sulphate and freezing tests is quite marked. It is noteworthy that no clay tile with an absorption of less than 7 percent was affected by 75 treatments by the accelerated freezing test or by twenty treatments by the sulphate test. For the type of tile tested, which were all made from shale, seven percent absorption would be a fairly safe maximum to insure resistance to freezing.

TABLE VII. ABSORPTION OF PIECES USED IN SODIUM SULPHATE TREATMENTS, A

PIECE NO.	ABSORPTION 72 HOURS IMMERSION	CLASS	PIECE NO.	ABSORPTION 72 HOURS IMMERSION	CLASS
1 E	9.85	16 in. Concrete, Factory L.....	34 D	7.66	
2 D	12.45		35 D	5.66	
3 D	8.71				
5 D	9.37		36 D	4.22	8 in. Clay Hard, Factory M...
6 F	7.74		37 D	4.74	
			38 E	4.64	
11 D	8.78	8 in. Concrete, Factory L....	39 D	6.68	
12 E	8.94		40 D	5.76	
13 D	9.15				
14 F	7.57		41 D	4.42	16 in. Clay Soft, Factory N...
15 F	7.95		42 D	4.66	
			43 D	4.50	
21 D	4.30	16 in. Clay Soft, Factory M....	44 D	4.27	
22 D	4.79		45 D	4.58	
23 D	2.72				
24 D	3.59		46 D	2.26	16 in. Clay Hard, Factory N...
25 E	4.94		47 D	4.02	
			48 D	2.36	
26 D	1.75	16 in. Clay Hard, Factory M...	49 F	3.23	
27 D	4.96		50 F	3.28	
29 D	3.69				
30 F	1.67		51 D	6.16	8 in. Clay Soft, Factory N...
			52 D	4.70	
31 D	7.94	8 in. Clay Soft, Factory M....	53 F	2.30	
32 E	7.24		54 D	7.36	
33 D	7.57		55 D	3.79	

TABLE VII — Concluded

PIECE NO.	ABSORPTION 72 HOURS IMMERSION	CLASS	PIECE NO.	ABSORPTION 72 HOURS IMMERSION	CLASS
56 D	2.96	8 in. Clay Hard, Factory N...	74 E	12.25	
57 D	2.67		75 D	14.02	
58 D	3.03				
59 F	2.58		76 D	9.52	8 in. Clay Hard, Factory P...
60 E	3.11		77 D	9.82	
			78 D	10.56	
61 D	9.40	16 in. Clay Soft, Factory P...	79 F	10.20	
62 D	11.45		80 F	8.62	
63 F	7.04				
64 D	9.20		81 D	5.49	12 in. Clay Soft, Factory R...
65 E	10.05		82 D	5.74	
			83 D	5.64	
66 D	10.61	16 in. Clay Hard (?), Factory P	84 D	5.57	
67 D	7.47		85 D	6.24	
68 D	8.68				
69 D	12.91		86 D	4.98	12 in. Clay Hard, Factory R...
70 E	7.59		87 D	5.45	
			88 E	5.71	
71 D	12.57	8 in. Clay Soft, Factory P....	89 D	5.32	
72 D	10.64		90 F	5.14	
73 D	13.60				

TABLE VIII. RESULTS OF SODIUM SULPHATE TREATMENTS, A, ON PIECES FROM TESTED TILE. FIVE TREATMENTS

16 hours in 15 percent Na_2SO_4 at 21° C. (70° F.), 7 hours in dryer at 110° C. (230° F.)

PIECE NO.	ABSORPTION 72 HOURS IMMERSION	FIRST NOTICEABLE EFFECT		EFFECT OF FIVE TREATMENTS, UNLESS SPECIMENS WERE DESTROYED BY A LESS NUMBER
		NUMBER OF TREATMENTS	DESCRIPTION OF EFFECT	
16 in. Concrete, Factory L				
1 E	9.85	2	1—½ in. spall	Cracked on edges, raw surfaces crumbling
2 D	12.45	2	2—½ in. spalls	Cracked on edges, raw surfaces crumbling
3 D	8.71	3	2—½ in. spalls	Cracked on edges, raw surfaces crumbling
5 D	9.37	3	1—½ in. spall	Cracked on edges, raw surfaces crumbling
6 F	7.74	3	1—½ in. spall off	Cracked on edges, raw surfaces crumbling
8 in. Concrete, Factory L				
11 D	8.78	3	1—½ in. spall	Badly cracked all through, raw surfaces crumbling
12 E	8.94	2	1—¾ in. spall	Badly cracked all through, raw surfaces crumbling
13 D	9.15	3	1—¾ in. spall, 1—½ in. crack	Destroyed, broke up in hands
14 D	7.57	5		Spall at one corner
15 F	7.95	2	1—½ in. spall	Badly cracked all through, raw surfaces crumbling

TABLE VIII — Concluded

16 hours in 15 percent Na_2SO_4 at 21° C. (70° F.), 7 hours in dryer at 110° C. (230° F.)

PIECE NO.	ABSORPTION 72 HOURS IMMERSION	FIRST NOTICEABLE EFFECT		EFFECT OF FIFTEEN TREATMENTS, UNLESS SPECIMENS WERE DESTROYED BY A LESS NUMBER
		NUMBER OF TREATMENTS	DESCRIPTION OF EFFECT	
		8 in. Clay Soft, Factory P		
71 D	12.57	2	1 spall on corner.....	Surface spalling at corners
72 D	10.64	5	.....	Slight spalling at corners
73 D	13.60	3	5—$\frac{1}{4}$ in. spalls.....	Spalled along 1 edge outside and 2 edges inside
74 E	12.25	3	3—$\frac{1}{4}$ in. spalls.....	Spalled along all edges outside and 1 edge inside
75 D	14.02	3	2 edges spalled in $\frac{1}{4}$ in. strips.....	Spalled along all edges outside and 2 edges inside
		8 in. Clay Hard, Factory P		
78 D	10.56	5	.....	4 $\frac{1}{4}$ in. spalls
79 C	10.20	5	.....	1 $\frac{1}{2}$ in. spalls at corner
		Other samples not affected		

TABLE IX. RESULTS OF SODIUM SULPHATE TREATMENTS, A, ON PIECES FROM TESTED TILE. TEN TREATMENTS

16 hours in 15 percent Na_2SO_4 at 21° C. (70° F.), 7 hours in dryer at 110° C. (230° F.)

PIECE NO.	ABSORPTION 72 HOURS IMMERSION	FIRST NOTICEABLE EFFECT		EFFECT OF TEN TREATMENTS, UNLESS SPECIMENS WERE DESTROYED BY A LESS NUMBER
		NUMBER OF TREATMENTS	DESCRIPTION OF EFFECT	
16 in. Concrete Factory L.				
1 E	9.85	2	1—½ in. spall	Destroyed by 8, broke in hands
2 D	12.45	2	2—½ in. spalls	Destroyed by 8, broke in hands
3 D	8.71	3	2—½ in. spalls	Destroyed by 8, broke in hands
5 D	9.37	3	1—½ in. spall	Destroyed by 7, broke in hands
6 F	7.74	3	1—½ in. spall	Destroyed by 8, broke in hands
8 in. Concrete Factory L.				
11 D	8.78	3	1—½ in. spall	Destroyed by 8, broke in hands
12 E	8.94	2	1—¾ in. spall	Destroyed by 8, broke in hands
13 D	9.15	3	1—¾ in. spall, 1—½ in. crack	Destroyed by 7, broke in hands
14 D	7.57	5	Spall at 1 corner	Softening around edges, breaks off in large chunks
15 F	7.95	2	1—½ in. spall	Destroyed by 8, broke in hands
8 in. Clay Soft, Factory M				
31 D	7.94	10		1 ¼ in. spall, old crack opening
34 D	7.66	10		Crumbling a little along 1 edge

TABLE IX — Concluded

16 hours in 15 percent Na_2SO_4 at 21° C. (70° F.), 7 hours in dryer at 110° C. (230° F.)

PIECE NO.	ABSORPTION 72 HOURS IMMERSION	FIRST NOTICEABLE EFFECT		EFFECT OF TEN TREATMENTS, UNLESS SPECIMENS WERE DESTROYED BY A LESS NUMBER
		NUMBER OF TREATMENTS	DESCRIPTION OF EFFECT	
8 in. Clay Soft, Factory N				
54 A	7.36	10	.....	Splitting along laminations 2 ¼ in. spalls
16 in. Clay Soft, Factory P				
61 D	9.40	8	Softening in 1 spot.....	Spalling a little around edges
62 D	11.45	8	1—½ in. spall.....	Spalling considerable around edges
63 F	7.04	10	.....	½ in. spall on 1 corner
64 D	9.20	10	.....	2 ½ in. spalls outside
65 E	10.05	8	3—¼ in. spalls.....	8 ¼ in. spalls
16 in. Clay Hard, Factory P				
66 D	10.61	10	.....	1 ¼ in. spalls
69 D	12.91	8	Spalling along 1 edge.....	Spalled badly along 2 edges

8 in. Clay Soft, Factory P

71 D	12.57	2	1 spall on corner	Destroyed by spalling
72 D	10.64	5	Slight spalling at corners	Badly spalled
73 D	13.60	3	5—¼ in. spalls	Destroyed, broke up in hands
74 E	12.25	3	3—¼ in. spalls	Almost all outside surface spalled off; 3 spalls inside
75 D	14.02	3	2 edges spalled in ¼ in. strips	Destroyed, broke up in hands

8 in. Clay Hard, Factory P

76 D	9.52	8	Small spall at edge	Spalled considerably in 1 spot
77 D	9.82	10		1 ¼ in. spall
78 D	10.56	5	4—¼ in. spalls	Badly spalled; 1/3 of sample gone
79 D	10.20	5	1—½ in. spall at corner	Spall increasing in size

Other samples not affected

TABLE X. RESULTS OF SODIUM SULPHATE TREATMENTS, A, ON PIECES FROM TESTED TILE. FIFTEEN TREATMENTS

16 hours in 15 percent Na_2SO_4 at 21° C. (70° F.), 7 hours in dryer at 110° C. (230° F.)

PIECE NO.	ABSORPTION 72 HOURS IMMERSION	FIRST NOTICEABLE EFFECT		EFFECT OF FIFTEEN TREATMENTS, UNLESS SPECIMENS WERE DESTROYED BY A LESS NUMBER
		NUMBER OF TREATMENTS	DESCRIPTION OF EFFECT	
16 in. Concrete, Factory L				
1 E	9.85	2	1—½ in. spall	Destroyed by 8
2 D	12.45	2	2—½ in. spalls	Destroyed by 8
3 D	8.71	3	2—½ in. spalls	Destroyed by 8
5 D	9.37	3	1—½ in. spall	Destroyed by 7
6 F	7.74	3	1—½ in. spall	Destroyed by 8
8 in. Concrete, Factory L				
11 D	8.78	3	1—½ in. spall	Destroyed by 8
12 E	8.94	2	1—¾ in. spall	Destroyed by 8
13 D	9.15	3	1—¾ in. spall, 1—½ in. crack	Destroyed by 7
14 D	7.57	5	Spall at 1 corner	Destroyed by softening
15 F	7.95	2	1—½ in. spall	Destroyed by 8
8 in. Clay Soft, Factory M				
31 D	7.94	10	1—¼ in. spall, old crack opening	Spalled in strip ½ in. wide along 3 edges; broke in two along old crack, ½ of sample out.
32 E	7.24	12	1—¼ in. spall	3 ¼ in. spalls
34 D	7.66	10	Crumbling a little along 1 edge	Spalled ⅛ in. along 1 edge

8 in. Clay Soft, Factory N				
54 D	7.36	10	Splitting along laminations; 2—¼ in. spalls	Spalled ⅛ to ¼ in. deep in 1 in. strip along 2 edges
16 in. Clay Soft, Factory P				
61 D	9.40	8	Softening in 1 spot	3 spalls
62 D	11.45	8	1 spall	Spalling along edges pronounced
63 F	7.04	10	¼ in. spall on 1 corner	Spall increased to ½ in.
64 D	9.20	10	2—½ in. spalls outside	5 ½ in. spalls
65 E	10.05	8	3—½ in. spalls	7 ½ in. spalls
16 in. Clay Hard, Factory P				
66 D	10.61	10	1 small spall	Spall increasing in size, 1 small spall.
69 D	12.91	8	Spalling along 1 edge	Destroyed, split all to pieces along laminations
8 in. Clay Soft, Factory P				
71 D	12.57	2	1 spall on corner	Destroyed by 10
72 D	10.64	5	Slight spalling at corners	Badly spalled, 1 corner broken off
73 D	13.60	3	5—¼ in. spalls	Destroyed, split to pieces along lamination
74 E	12.25	3	3—¼ in. spalls	Edges breaking off, 50 percent of surface gone
75 D	14.02	3	2 edges spalled in ¼ in. strips	Destroyed in 10
8 in. Clay Hard, Factory P				
76 D	9.52	8	1—¼ in. spall at edge	Badly spalled
77 D	9.82	10	1—¼ in. spall	4 ¼ in. spalls
78 D	10.56	5	4—¼ in. spalls	All outer surface and ¾ of inner surface gone
79 F	10.20	5	1—½ in. spall at corner	Spall on corner increased to ¾ by 1 in.
Other samples not affected				

TABLE XI. RESULTS OF SODIUM SULPHATE TREATMENTS, A, ON PIECES FROM TESTED TILE. TWENTY TREATMENTS

16 hours in 15 percent Na_2SO_4 at 21° C. (70° F.), 7 hours in dryer at 110° C. (230° F.)

PIECE NO.	ABSORPTION 72 HOURS IMMERSION	FIRST NOTICEABLE EFFECT		EFFECT OF TWENTY TREATMENTS, UNLESS SPECIMENS WERE DESTROYED BY A LESS NUMBER
		NUMBER OF TREATMENTS	DESCRIPTION OF EFFECT	
16 in. Concrete, Factory L				
1 E	9.85	2	1—½ in. spall	Destroyed by 8
2 D	12.45	2	2—½ in. spalls	Destroyed by 8
3 D	8.71	3	2—½ in. spalls	Destroyed by 8
5 D	9.37	3	1—½ in. spall	Destroyed by 7
6 F	7.74	3	1—½ in. spall	Destroyed by 8
8 in. Concrete, Factory L				
11 D	8.78	3	1—½ in. spall	Destroyed by 8
12 E	8.94	2	1—¾ in. spall	Destroyed by 8
13 D	9.15	3	1—¾ in. spall, 1—½ in. crack	Destroyed by 7
14 D	7.57	5	Spall at 1 corner	Destroyed by softening
15 F	7.95	2	1—½ in. spall	Destroyed by 8

8 in. Clay Soft, Factory M

31 D	7.94	10	1—¼ in. spall, old crack opening	⅜ of outer surface gone
32 E	7.24	12	1—¼ in. spall	5 ¼ in. spalls
33 D	7.57	18	2—¾ in. spalls inside	Spalled for ½ in. along 3 edges outside; 2 ½ by ¾ in. spalls inside
34 D	7.66	10	Crumbling a little along 1 edge	⅓ of outer surface and ⅓ of inner surface gone; ½ by ½ by 2 in. chunk off 1 side

8 in. Clay Soft, Factory M

54 D	7.36	10	Splitting along laminations; 2—¼ in. spalls	Destroyed in 18, broke all to pieces along laminations

16 in. Clay Soft, Factory P

61 D	9.40	8	Softening in 1 spot	5 ½ in. spalls off edge
62 D	11.45	8	1 spall	Surface spalled off 3 edges outside, 5 ¼ in. spalls inside
63 F	7.04	10	1—¼ in. spall on corner	6 ¼ in. spalls
64 D	9.20	10	2—½ in. spalls outside	2 ¼ by ½ in. and 2 ¼ in. spalls
65 E	10.05	8	3—½ in. spalls	Spalled in ¼ in. strip along 3 edges inside and 1 outside

16 in. Clay Hard, Factory P

66 D	10.61	10	1—¼ in. spall	4 ¾ in. spalls
69 D	12.91	8	Spalling along 1 edge	Destroyed in 15

TABLE XI — Concluded

16 hours in 15 percent Na_2SO_4 at 21° C. (70° F.), 7 hours in dryer at 110° C. (230° F.)

PIECE NO.	ABSORPTION 72 HOURS IMMERSION	FIRST NOTICEABLE EFFECT		EFFECT OF TWENTY TREATMENTS, UNLESS SPECIMENS WERE DESTROYED BY A LESS NUMBER
		NUMBER OF TREATMENTS	DESCRIPTION OF EFFECT	
8 in. Clay Soft, Factory P				
71 D	12.57	5	Surface spalling at corners	Destroyed by 10
72 D	10.64	5	Slight spalling of surface at corners.	$\frac{3}{4}$ of outer surface and $\frac{1}{2}$ of inner surface gone
73 D	13.60	3	5—$\frac{1}{4}$ in. spalls	Destroyed in 15
74 E	12.25	3	3—$\frac{1}{4}$ in. spalls	Destroyed in 18, by spalling
75 D	14.02	3	2 edges spalled in $\frac{1}{4}$ in. strip	Destroyed in 10
8 in. Clay Hard, Factory P				
76 D	9.52	8	1—$\frac{1}{4}$ in. spall at edge	Surface along 1 edge gone, 6 other spalls
77 D	9.82	10	1—$\frac{1}{4}$ in. spall	5 $\frac{1}{4}$ in. spalls
78 D	10.56	5	4—$\frac{1}{4}$ in. spalls	Destroyed by spalling
79 F	10.20	5	1—$\frac{1}{2}$ in. spall at corner	1 $\frac{3}{4}$ by 1$\frac{1}{4}$ and 2 $\frac{1}{4}$ in. spalls
80 F	8.62	18	1—$\frac{1}{4}$ in. spall	Strip $\frac{3}{4}$ by 2$\frac{1}{2}$ spalled off edge
Other samples not affected				

Method B. The absorptions of the samples used in this series of tests are shown in Table XII and the results in Tables XIII and XIV. The results are similar to those obtained by Method A. However, this method of testing is less severe than Method A and gives results much more slowly. It is, therefore, inferior to Method A.

TABLE XII. ABSORPTION OF PIECES USED IN SODIUM SULPHATE TREATMENTS, B

PIECE NO.	ABSORPTION 72 HOURS IMMERSION	CLASS	PIECE NO.	ABSORPTION 72 HOURS IMMERSION	CLASS
1 F	8.55	16 in. Concrete, Factory L.	51 E	6.24	8 in. Clay Soft, Factory N.
2 E	9.68		52 E	4.27	
11 E	8.20	8 in. Concrete, Factory L.	56 E	2.95	8 in. Clay Hard, Factory N.
12 F	8.10		57 E	2.61	
21 E	5.59	16 in. Clay Soft, Factory M.	61 E	9.21	16 in. Clay Soft, Factory P.
22 F	4.96		62 E	10.25	
26 E	1.94	16 in. Clay Hard, Factory M.	66 E	10.77	16 in. Clay Hard, Factory P.
27 E	4.76		67 E	6.94	
31 E	7.64	8 in. Clay Soft, Factory M.	71 E	13.02	8 in. Clay Soft, Factory P.
32 F	7.65		72 E	10.76	
36 E	4.58	8 in. Clay Hard, Factory M.	76 E	9.24	8 in. Clay Hard, Factory P.
37 E	4.47		77 E	10.57	
41 E	4.54	16 in. Clay Soft, Factory N.	81 E	5.62	12 in. Clay Soft, Factory R.
42 F	4.59		82 E	6.07	
46 E	2.25	16 in. Clay Hard, Factory N.	86 F	4.72	12 in. Clay Hard, Factory R.
47 E	7.26		87 E	5.70	

TABLE XIII. RESULTS OF SODIUM SULPHATE TREATMENTS, B, ON PIECES FROM TESTED TILE. FIVE TREATMENTS

24 hours in 16 percent Na_2SO_4 at 70° F., 24 hours in room at 70° F.

PIECE NO.	ABSORPTION 72 HOURS IMMERSION	FIRST NOTICEABLE EFFECT		EFFECT OF FIVE TREATMENTS, UNLESS SPECIMENS WERE DESTROYED BY A LESS NUMBER
		NUMBER OF TREATMENTS	DESCRIPTION OF EFFECT	
16 in. Concrete, Factory L				
1 F	8.55	4	Crumbling at corners and edges....	Crumbles at corners and edges
2 E	9.68	3	Cracked in 3 places, raw surfaces crumbling	Badly cracked, crumbles at edges
8 in. Concrete, Factory L				
11 E	8.20	3	Edges break off in hands.....	Edges break off in hands
12 F	8.10	3	Cracked in two, one part crumbles in hands	Destroyed by disintegration

Other samples not affected

TABLE XIV. RESULTS OF SODIUM SULPHATE TREATMENTS, B, ON PIECES FROM TESTED TILE. TEN TREATMENTS

24 hours in 16 percent Na_2SO_4 at 70° F., 24 hours in room at 70° F.

PIECE NO.	ABSORPTION 72 HOURS IMMERSION	FIRST NOTICEABLE EFFECT		EFFECT OF TEN TREATMENTS, UNLESS SPECIMENS WERE DESTROYED BY A LESS NUMBER
		NUMBER OF TREATMENTS	DESCRIPTION OF EFFECT	
16 in. Concrete, Factory L				
1 F	8.55	4	Crumbling at corners and edges.....	Disintegrated in hands
2 E	9.68	3	Cracked in 3 places, raw surfaces crumbling	Destroyed in 6, by disintegration
8 in. Concrete, Factory L				
11 E	8.20	3	Edges break off in hands.....	Crumbling badly along edges
12 F	8.10	3	Cracked in two, one part crumbles in hands	Destroyed in 5
16 in. Clay Hard, Factory P				
66 E	10.77	6	One small spall.....	Spall a little larger
8 in. Clay Soft, Factory P				
72 E	10.76	6	One spall at corners.....	Spall larger

Other samples not affected

Method C. The absorptions of the samples used in this series of tests are shown in Table XV, and the results are summarized in Table XVI. This test demonstrated conclusively that on some samples of concrete pipe, sodium sulphate in solution has a decided action other than that due to the crystallization of some form of the salt. Of course we would expect $Ca\ SO_4 . 2\ H_2O$ to be formed, but cement chemists claim that highly hydrated compounds of lime alumina and SO_4 are also formed. In fact the disintegration of concrete in sea water is attributed to the action of soluble sulphates.

The following is a quotation from an article by Dr. Wilhelm Michaelis.[5]

"In Portland cement the hardened mass is completely permeated with crystals of calcic hydrate. The hydrate of the combined aluminate and sulphate of lime (Al_2O_3, $3CaO + 3CaOSO_3 + 30\ H_2O$) has been observed when the compound was dried over sulphuric acid.

"A body containing a substance of such strong chemical affinity as free lime cannot be regarded as stable. The free lime will continue to react until it forms a saturated compound. When the mortar hardens in the air or in the water containing carbonic acid the lime is converted into carbonate; but in sea-water it is chiefly the sulphates which act upon the lime. In the first instance it is the free lime that is converted into carbonate or sulphate, then the very unstable compound with ferric oxide, then the aluminate, and finally the silicate. The formation of the sulphate with two equivalents of water causes a considerable increase of volume, and may destroy the cohesion of the mass. With this formation of gypsum the production of lime aluminate-sulphate goes hand in hand, and this causes an enormous increase of volume, and a total destruction of the cohesion, for this double compound crystallizes with at least 30, probably with 60 equivalents of water, and in doing so converts the strongest mortar into a mud, the only parts of which retaining any cohesion being those protected by the formation of carbonate."

Following is a statement by Le Chatelier.[6]

"When the calcium sulphate found in natural waters or formed by the interaction of magnesium sulphate and the calcium compounds of cement reacts with calcium aluminate it produces a calcium sulphoaluminate whose crystallization gives rise to swelling and cracking in the material. The action resembles that consequent upon the hydration of quicklime, but is much slower."

[5] *Proceedings of the Institution of Civil Engineers*, Vol. CXXIX, 1896, pages 325-6.

[6] *Congress of the International Association for Testing Materials*, Brussels, 1906.

From the standpoint of the cement chemist, the chemical action of soluble sulphates on concrete would depend: on the composition of the cement, on the density of the concrete, and especially on the extent to which it is protected by insoluble carbonates. It is noteworthy that in this test the raw edges of the samples were attacked first, and that the thin samples stood the tests better than the thick.

That the results for the concrete samples in test A are so closely comparable to the freezing tests is probably due to the severe mechanical strains set up in this test by the crystallization of $Na_2\ SO_4$. These would tend to obscure the effects of the lesser strains due to the formation of new chemical compounds of large volume. However, sodium sulphate tests can not be considered a reliable substitute for freezing tests on concrete.

TABLE XV. ABSORPTION OF PIECES USED IN SODIUM SULPHATE TREATMENTS, C

PIECE NO.	ABSORPTION 72 HOURS IMMERSION	CLASS	PIECE NO.	ABSORPTION 72 HOURS IMMERSION	CLASS
3 E	7.37	16 in. Concrete, Factory L.	58 E	2.99	8 in. Clay Hard, Factory N.
5 E	10.27		59 E	2.73	
13 F	7.55	8 in. Concrete, Factory L.	63 E	7.12	16 in. Clay Soft, Factory P.
14 E	7.65		64 E	8.70	
23 E	3.22	16 in. Clay Soft, Factory M.	68 E	7.92	16 in. Clay Hard, Factory P.
24 E	3.59		69 E	12.70	
29 E	2.32	16 in. Clay Hard, Factory M.	73 E	12.78	8 in. Clay Soft, Factory P.
33 E	7.72	8 in. Clay Soft, Factory M.	74 F	12.85	
34 F	7.62		78 E	10.70	8 in. Clay Hard, Factory P.
38 F	4.26	8 in. Clay Hard, Factory M.	79 E	10.45	
39 E	6.80		83 E	5.52	12 in. Clay Soft, Factory R.
43 F	4.24	16 in. Clay Soft, Factory N	84 E	5.62	
44 E	3.85		88 F	5.21	12 in. Clay Hard, Factory R.
48 E	2.40	16 in. Clay Hard, Factory N.	89 E	5.54	
49 E	2.77				
53 E	2.83	8 in. Clay Soft, Factory N.			
54 F	6.89				

TABLE XVI. RESULTS OF SODIUM SULPHATE TREATMENTS, C, ON PIECES FROM TESTED TILE. TWENTY DAYS

Continuous immersion in 15 percent Na_2SO_4 at 70° F.

PIECE NO.	ABSORPTION 72 HOURS IMMERSION	FIRST NOTICEABLE EFFECT		EFFECT OF TWENTY DAYS' IMMERSION
		NUMBER OF TREATMENTS	DESCRIPTION OF EFFECT	
16 in. Concrete, Factory L				
3 E	7.37	5	Three cracks, raw surface softening in spots	Destroyed in 14, broke up in hands
5 E	10.27	5	Raw surface softening in spots	Destroyed, most of sample has crumbled away, rest breaks up in hands
8 in. Concrete, Factory L				
13 F	7.55	10	Softening a little at corners	Crumbling along edge and at corners
14 E	7.65	10	Softening a little at corners	Crumbling along edges

Other samples not affected

GENERAL CONCLUSIONS

I. The method of conducting freezing tests by the partial-immersion process, described under Accelerated Freezing Tests, Second Series, is the quickest and most satisfactory of those tried.

II. The method of conducting sodium sulphate tests, described under Sodium Sulphate Tests, Method A, is quicker, more convenient, and more economical than any of the freezing tests or other sulphate tests tried. One treatment by this method is approximately equal to two treatments by the partial-immersion freezing method.

III. The parallelism between high porosity and easy attack by both freezing and sulphate tests is quite marked. Seven per-cent would seem a safe maximum absorption for clay tile of the type tested (made from shale clay).

IV. Due to the chemical action of soluble sulphates on concrete, sodium sulphate tests can not be considered a reliable substitute for freezing tests on concrete tile. See Sodium Sulphate Tests, Method C.

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PYROMETRY: PAST, PRESENT AND FUTURE

BY EDWARD C. STOVER, TRENTON, N. J.

Pyrometry is a many phased subject.[1]

The first pyrometric method was introduced by Sir. Isaac Newton. It was a bar of iron which lengthened on heating and this increase was taken as a measure of the temperature increase. The method was published in 1701.

The first practical instrument was designed by Josiah Wedgewood in 1782. This is a ceramic block which shrinks on heating, and its shrinkage is an indicator of the temperature attained. This instrument was without a rival for 40 years and is still employed in the pottery district of Staffordshire, England.

In 1822 John Daniel introduced the platinum bar encased in a plumbago cover upon which was a scale to show the expansion of the platinum. This increase was read by means of a telescope. In the same year Seebecks discovered the principle of thermo-electrics. He found that a current of electricity is generated by heating a junction of two metals, platinum and palladium.

In 1830 Becquerel built an instrument of this type, but it was practically abandoned, and not until 1886 was it revived in a more practical form, and led to the present extensive use of pyrometers.

Dr. Herman A. Seger published his invention of Seger Pyrometric Cones in 1886 in Berlin, Germany, and their use in both Europe and the United States continues to the present time.

Even in the early days of pyrometry, each method was recognized as having special advantages in particular cases. Many potters, being ignorant of the particular adaptability of the different methods have chosen a method not adapted to their needs

[1] *History of Pyrometry*, by Chas. R. Darling, published by Spon, London, 1911.

and hence have received more or less indifferent aid by its use. They have therefore condemned pyrometry in general.

The experienced potter is becoming more and more impressed with the importance of temperature changes on his ware in the kiln, from the begining to the finish and also in the rate of cooling. The time is fast approaching when combinations of the methods of pyrometry will be employed by the potter, so that he will know the condition of the various parts of his kiln, from start to finish, including cooling in somewhat the same manner as the metallurgist has done. A great many metals are not only sold under specifications as to chemical compositions, but also as to the process of preparation and heat treatment necessary to insure definite results that can be accurately duplicated at any time.

The Present in Ceramics seems to be one of education. With the proper combination including the recording pyrometer from start to finish a great saving of fuel is possible, to say nothing of eliminating losses and producing a more uniform product.

The Solon shrinker rings are now being used by some people. This method necessitates withdrawing the ring from the kiln every 3 or 4 hours and taking actual measurement thereof. They have been found quite accurate, but they do not cover the time as closely as it really should be covered. This is admitted by many who are using them.

The Future. The time is fast approaching when the experienced fireman will refuse a position where recording pyrometers have not been installed to give him a chance to prove his claims of accuracy and economy in firing.

A COMPARISON BETWEEN THE ABSORPTION, CRUSHING STRENGTH AND RESISTANCE TO ARTIFICIAL FREEZING OF SOME OHIO BUILDING BRICKS

BY EDWARD ORTON, JR.

In connection with the work of Committee C-3 of the American Society for Testing Materials, which concerns itself with the properties of and specifications for bricks, an effort has been made to collect data from all available sources in the United States on the physical properties of the bricks used in building work, not including paving bricks, fire bricks, hollow bricks or other special varieties. The data sought chiefly has related to absorption percent, crushing strength and cross breaking strength or modulus of rupture, but attention has been called to the importance of the freezing test. It has been stated that the other strength tests are all of them indirect and inconclusive, and that if we were able to completely assure ourselves of the weather resisting ability of building bricks, the need of making any other tests would be very largely obviated. The difficulty of making the freezing test rapidly and at any time or place is so considerable, and requires the use of such expensive equipment, that actual use of the test is infrequent thus far, and there does not seem any great likelihood of its extensive use in the near future.

In Volume XVII., *Transactions of the American Ceramic Society*, page 249, W. L. Howat of the U. S. Bureau of Standards, published a valuable paper on the use of the sodium sulphate solution as a mode of producing artificially, and at convenient temperatures, the destructive effect of the freezing of water in the pore spaces of brick. The method and interesting results obtained by Mr. Howat can be readily consulted.

The importance of the sodium sulphate process, if it is found to offer a really practical substitute for ice freezing, is

great, and no pains should be spared to work out and bring into public notice the exact relationship between the two. If it is found, as is foreshadowed by Mr. Howat's paper and the discussion upon it, that the action of sodium sulphate is similar in kind, but much more severe than that of ice, it should be possible to establish such a ratio in the number of treatments given, or to so regulate the exact conditions of the test as to get a modified or partial action, the results of which would be equivalent to ice freezing.

The present investigation was begun primarily to fill a gap in the records of Committee C-3 regarding the relative crushing strength exhibited by building bricks when crushed upon their flat compared to that when crushed on their edge. To make such a comparison valuable, the material tested by the two methods must be as nearly the same as possible. It was necessary to not only compare bricks of the same make, but of the same hardness or degree of vitrification. This involved securing a good stock of each material used in the test, and making a careful absorption test upon every brick, and then selecting for the comparison, groups of bricks whose individual members should only vary between narrow limits above and below the average.

To broaden this plan to include a study of the behavior of the same material under the sodium sulphate freezing test, seemed highly desirable from several points of view. The Howat investigation had not been conducted upon commercial bricks, but upon laboratory-made briquettes only, and upon relatively few specimens. Also freezing tests by either ice or sodium sulphate would be unsatisfactory and inconclusive during the preliminary or investigational phase of the process, if not accompanied by concurrent information on the strength and other properties of the material under study. The original plan was therefore modified by providing enough material of each kind to make two accurately graded sets of specimens of the same average absorption, using one for crushing in the two positions, and the other for concurrent tests of resistance to disintegration by sodium sulphate freezing.

The Samples. The samples were furnished gratuitously by various brick manufacturers in Ohio. The considerations governing the request for the furnishing of samples were as follows:

1. To cover the state geographically as well as could be done with a limited number of samples.

Fourteen samples were obtained representing the following districts:

Northeastern quarter — Cleveland, Sandusky	3
Southeastern quarter — Zanesville, Somerset, Junction City	4
Central — Columbus, Taylor Station	2
Southwestern quarter — Cincinnati, Delphi, Dayton	5
Northwestern quarter — Toledo	2
	14

2. To represent the large producers of building brick, rather than those who make paving brick their chief product, producing building brick as a side issue.

In the former category belong the plants representing Cleveland, Sandusky, Toledo, Cincinnati, Dayton and Columbus	12
In the latter belong Zanesville and Junction City	2
	14

3. To represent the different types of materials used in Ohio building brick industries.

The samples are classified as representing glacial surface clays.

Sandusky	2	
Toledo	1	
Columbus	1	
Dayton	1	
Delphi (Cincinnati)	1	
		6
Alluvial clays —		
Cincinnati	1	
		1

Shale clays —		
Taylor Station	1	
Junction City	1	
Somerset	1	
Zanesville	1	
Cleveland	1	
		5
Sand-lime brick	1	
Cement-sand brick	1	
		2
		14

4. To represent the different processes of brick manufacture.

The samples were divided as follows:

Clay, stiff-mud, auger machine product	7
Clay, soft-mud, sand mold machine product	5
Sand-lime process	1
Cement-sand process	1
	14

Enquiry for a dry-press common building brick was made, but no plant now using this process could be located at the time.

5. In addition to the above considerations, it was desired to fairly represent the entire product of each firm furnishing samples, and accordingly each was requested to furnish three grades of material:

1 — The hardest burnt material which they habitually sell.
2 — Their regular or average product.
3 — The softest material which they habitually sell.

This really made thirty-four (34) separate samples, of fourteen different makes. The two hydraulic-process bricks were represented by one grade each, and some of the clay brick makers sent only two instead of three grades.

6. The number of bricks required of each grade was twelve, which were apportioned for test as follows:

To the absorption test	All
To the crushing test	5
To the sodium sulphate test	5
For reserve	2
	12

The total number of different bricks used in all the tests was approximately 432.

7. For obvious reasons, the names of manufacturers who sent samples are withheld.

CLASSIFICATION OF THE MATERIAL IN GROUPS BY THE ABSORPTION TEST

Drying Out. Each lot of bricks was cleaned by brushing, each brick being given a number in lead pencil, and then placed in a muffle kiln, and heated for an hour or two at 110-120° C. They were then weighed individually and returned to the kilns, heated for an hour or two to 110-120° C. and again weighed. If second weight now confirmed the first, it was assumed that the bricks were dry. If not, a third drying treatment was given, or until a check weight was obtained.

Weighing. The bricks were weighed on a special balance, reading in hundredths of pounds.

Soaking. The bricks were placed in a metal tank and covered with water. This was heated to boiling and boiled about four hours, and the bricks allowed to cool down in the water.

Reweighing. When cool the bricks were taken from the water, allowed to drain five minutes, and then weighed. The difference between the wet and dry weight was then taken, and the percentage calculated in terms of the dry weight.

Redrying. The bricks were now placed in drying ovens described later, and heated to 110-120° C. for from twenty to twenty-four hours, at the end of which time they were found to be entirely dry again.

Grouping. Each material was now classified into groups of five bricks each, the mean absorption of each pair of groups

being as nearly identical as possible. The extra bricks were set aside for reserve. For illustration, the data used in grouping Brick A is given in full. See Table I.

TABLE I — SAMPLE NO. 1

BRICK NUMBER	COMMERCIAL GRADING	ABSORPTION
		percent
1		6.37
2		5.14
3		6.49
4		5.90
5		5.32
6	Hard	4.40
7		4.33
8		5.32
9		6.41
10		4.31
11		5.23
12		6.14
13		12.57
14		15.68
15		13.88
16		14.66
17		13.89
18	Medium	12.62
19		14.26
20		12.85
21		12.80
22		12.55
23		13.12
24		13.10
25		13.57
26		15.35
27		18.92
28		13.52
29		18.64
30		18.77
31	Soft	14.61
32		17.86
33		17.71
34		17.04
35		18.33
36		18.40

From these 36 bricks the groups as shown in Table II were selected.

TABLE II — SHOWING ARRANGEMENT OF SAMPLE BRICKS INTO PAIRS OF GROUPS OF EQUAL ABSORPTION GRADE

GRADE	FOR CRUSHING		FOR FREEZING		FOR RESERVE	
	No.		*No.*		*No.*	
Hard.	10	4.31 Min.	7	4.33 Min.		
	6	4.40	2	5.14		
	5	5.32	11	5.23	9	6.41
	4	5.90	8	5.32	3	6.49
	1	6.37 Max.	12	6.14 Max.		
		5.26 Average		5.23 Average		
Medium.	22	12.55 Min.	18	12.62 Min.		
	20	12.85	21	12.80	12	12.57
	23	13.12	24	13.10	16	14.66
	28	13.52	25	13.57		
	17	13.89 Max.	15	13.88 Max.		
		13.19 Average		13.19 Average		
Soft.	14	15.68 Min.	26	15.35 Min.		
	34	17.04	33	17.71	19	14.26
	35	18.33	32	17.86	31	14.61
	36	18.40	29	18.64		
	27	18.92 Max.	30	18.77 Max.		
		17.67 Average		17.66 Average		

This preliminary work of grading and classification of the fourteen sets of samples by the absorption test into as nearly as possible strictly comparable groups was a labor of some magnitude, and occupied about a month's time.

CRUSHING TESTS

To remove so far as possible variations due to unequalities in the test pieces themselves, each brick was divided into two equal parts, one-half being crushed flatwise and the other half on edge. To avoid weakening the structure by shock, each brick was sawed to a depth of about one-fourth inch on all four sides on the center line, using a one-eighth vulcanite wheel. A blow was then given with a mason's chisel of four and one-half inches width of face, at the weakened center zone. The bricks usually broke with a square clean fracture, with almost no chip-

ping or spalling. If the break was bad, it was smoothed up lightly on a coarse emery wheel to remove the protruding bumps. In this way, two test pieces of very nearly equal size and value were obtained from each brick.

The details of the crushing test were the usual ones as prescribed by the preliminary standard methods of Committee C-3 and need not be enlarged upon here. The test pieces were given a coat of shellac on the top and bottom surfaces and then trued up with plaster of Paris, hardened between parallel pieces of plate glass, previously oiled. The machine used was a 200,000 pound Olsen. The five tests on each group, by each method, were then averaged and the results compared.

These results are set forth in the Tables, III to XV inclusive.

TABLE III — SAMPLE NO. 1

Process of Manufacture..... Stiff Mud, Side Cut
Kind of Clay Used..... Shale.

ABSORPTION	CRUSHING ON EDGE	CRUSHING ON FLAT	RATIO EDGE: FLAT
Group 1 — Hard			
4.31	7530	12780	
4.40	9923	15340*	
5.32	11200	12260	
5.90	9820	10625	
6.37	6250	10485	
5.26	8945	12298	1 : 1.37
Group 2 — Medium			
12.55	5076	6105	
12.85	5504	7575	
13.12	4903	6290	
13.52	4478	7455	
13.89	3506	5243	
13.19	4693	6554	1 : 1.39
Group 3 — Soft			
15.68	2976	2984	
17.04	3271	3866	
18.33	2709	2829	
18.40	2031	3094	
18.92	1914	2668	
17.67	2580	3088	1 : 1.19

* Not crushed at max. pressure available.

TABLE IV — SAMPLE NO. 2

Process of Manufacture..... Stiff Mud, Side Cut.
Kind of Clay Used..... Shale.

ABSORPTION	CRUSHING ON EDGE	CRUSHING ON FLAT	RATIO EDGE: FLAT
Group 1 — Hard			
1.47	9700	13460*	
1.97	7230	13460*	
1.97	8535	13810*	
2.27	8184	12810*	
2.60	4646	12770	
2.06	7659	13262	1 : 1.73
Group 2 — Medium			
2.72	13940	12360	
2.72	11548	13390	
3.28	13674	13815*	
4.01	9815	13814*	
4.39	7438	11595	
3.42	11283	13001	1 : 1.15
Group 3 — Soft			
6.28	7330	10152	
7.95	5679	8524	
8.10	5113	12212	
8.72	6969	12132	
8.98	4580	7651	
8.01	5934	10134	1 : 1.70

* Did not crush at max. available pressure.

* Did not crush at max. available pressure.

TABLE V — SAMPLE NO. 3

Process of Manufacture..... Soft Mud, Sand Mold Machine.
Kind of Clay Used..... Glacial, Surface Material.

ABSORPTION	CRUSHING ON EDGE	CRUSHING ON FLAT	RATIO EDGE : FLAT
		Group 1 — Hard	
18.82	2868	3496	
18.89	2376	3244	
19.30	3794	3347	
19.41	2819	3007	
19.92	2542	2978	
19.27	2880	3214	1 : 1.11
		Group 2 — Soft	
20.32	2623	2143	
21.28	1555	3517	
23.24	600	638	
24.55	396	902	
26.38	458	1000	
23.15	1126	1640	1 : 1.46

TABLE VI — SAMPLE NO. 4

Process of Manufacture..... Stiff Mud, End Cut.
Kind of Clay Used..... Alluvial.

ABSORPTION	CRUSHING ON EDGE	CRUSHING ON FLAT	RATIO EDGE: FLAT
Group 1 — Hard			
11.13	5253	6521	
11.65	5987	5983	
12.32	5297	6300	
12.85	4191	5356	
12.92	4407	5140	
12.17	5027	5860	1:1.16
Group 2 — Medium			
14.36	3475	4779	
15.96	3668	3287	
16.45	1524	3000	
18.25	3397	3731	
18.35	2108	4182	
16.67	2835	3796	1:1.34
Group 3 — Soft			
19.19	1500	2434	
19.64	2258	2469	
20.80	1572	2012	
21.69	1365	2147	
22.84	844	1311	
20.83	1508	2075	1:1.37

TABLE VII — SAMPLE NO. 5

Process of Manufacture..... Stiff Mud, Side Cut.
Kind of Clay Used..... Glacial, Surface Material.

ABSORPTION	CRUSHING ON EDGE	CRUSHING ON FLAT	RATIO EDGE: FLAT
		Group 1 — Hard	
14.31	6593	8622	
15.66	3142	7655	
16.22	5790	9150	
16.86	3048	6843	
17.74	6382	7463	
16.16	4991	7947	1:1.59
		Group 2 — Medium	
17.74	4086	6560	
17.91	5154	7213	
18.16	3683	9190	
18.21	4513	8221	
19.96	3120	7526	
18.39	4111	7742	1:1.88
		Group 3 — Soft	
20.79	2034	5121	
21.21	2101	3546	
21.48	2195	4005	
21.70	1622	3488	
21.97	1571	4101	
21.42	1905	4052	1:2.13

TABLE VIII — SAMPLE NO. 5-A

Process of Manufacture..... Soft Mud, Sand Mold Machine.
Kind of Clay Used..... Glacial, Surface Material.

ABSORPTION	CRUSHING ON EDGE	CRUSHING ON FLAT	RATIO EDGE: FLAT
		Group 1 — Hard	
11.26	5700	7045	
12.30	4088	6043	
13.17	4318	5505	
15.96	5223	4603	
17.07	5052	5513	
13.95	4876	5742	1:1.18
		Group 2 — Medium	
19.34	4439	4103	
20.41	3093	3404	
21.51	3712	4128	
22.90	2632	2500	
23.50	2810	3882	
21.53	3337	3603	1:1.08
		Group 3 — Soft	
24.79	2320	2538	
25.46	1750	2531	
26.07	1550	2149	
27.63	1409	2126	
29.39	1365	1944	
26.67	1679	2258	1:1.34

TABLE IX — SAMPLE NO. 6

Process of Manufacture..... Stiff Mud, Side Cut.
Kind of Clay Used..... Shale.

ABSORPTION	CRUSHING ON EDGE	CRUSHING ON FLAT	RATIO EDGE: FLAT
Group 1 — Hard			
1.67	9766	13150	
1.84	10156	14540*	
2.01	4530	11984	
3.71	10460	13470*	
3.95	15685	12732	
2.63	10119	13175	1:1.30
Group 2 — Medium			
3.93	8965	10220	
5.64	10050	13960	
6.04	10100	13817	
7.02	10060	10734	
7.64	9774	10932	
6.05	9790	11932	1:1.22
Group 3 — Soft			
14.54	4966	5847	
15.76	4113	4234	
16.01	4347	4146	
16.73	2795	3822	
17.54	3088	3747	
16.11	3862	4359	1:1.13

* Not crushed at maximum pressure available.

TABLE X — SAMPLE NO. 7

Process of Manufacture..... Soft Mud, Sand Mold Machine.
Kind of Clay Used..... Glacial, Surface Material

ABSORPTION	CRUSHING ON EDGE	CRUSHING ON FLAT	RATIO EDGE: FLAT
		Group 1 — Hard	
16.16	3295	2963	
17.15	3130	3235	
17.85	2871	2551	
17.97	2748	2820	
18.38	3127	2820	
17.50	3034	2878	1:0.94
		Group 2 — Soft	
21.18	1642	1934	
21.20	1338	1819	
21.39	1360	1969	
21.54	1410	2273	
21.82	1356	1689	
21.43	1421	1937	1:1.36

TABLE XI — SAMPLE NO. 8

Process of Manufacture..... Stiff Mud, Side Cut.
Kind of Clay Used..... Shale.

ABSORPTION	CRUSHING ON EDGE	CRUSHING ON FLAT	RATIO EDGE : FLAT
		Group 1 — Hard	
1.00	15190	13438	
1.01	11788	13539	
1.16	14000	14139*	
1.31	16956	13547	
1.51	17760	11442	
1.19	15139	13232	1 : 0.87
		Group 2 — Medium	
5.69	10830	12865	
6.47	10402	12234	
6.62	10330	12550	
7.46	9904	12385	
7.52	12924	11267	
6.75	10878	12260	1 : 1.12
		Group 3 — Soft	
11.96	5596	7953	
12.07	6567	7206	
12.96	6133	5102	
13.20	5315	5888	
13.62	4737	5115	
12.75	5669	6253	1 : 1.10

* Not crushed at maximum pressure available.

TABLE XII — SAMPLE NO. 9

Process of Manufacture..... Sand-Lime.
Kind of Material Used..... Sand and Calcium Hydroxide.

ABSORPTION	CRUSHING ON EDGE	CRUSHING ON FLAT	RATIO EDGE: FLAT
11.65	2097	2718	
12.61	2156	2253	
12.66	1838	2775	
13.17	1865	2332	
13.24	1606	2780	
12.66	1912	2572	1:1.34

SAMPLE NO. 10

Process of Manufacture..... Soft Mud, Sand Mold Machine.
Kind of Clay Used..... Glacial, Surface Material

ABSORPTION	CRUSHING ON EDGE	CRUSHING ON FLAT	RATIO EDGE: FLAT
Group 1 — Hard			
17.39	3174	3323	
17.74	2890	2937	
17.96	777	779	
18.62	1138	1356	
19.39	2770	2596	
18.22	2168	2198	1:1.01
Group 2 — Soft			
20.24	1378	1975	
20.33	1986	1987	
20.53	1970	2415	
20.83	1900	1861	
21.06	2210	1721	
20.60	1889	1992	1:1.05

TABLE XIII — SAMPLE NO. 11

Process of Manufacture..... Soft Mud, Sand Mold Machine.
Kind of Clay Used..... Glacial, Surface Material

ABSORPTION	CRUSHING ON EDGE	CRUSHING ON FLAT	RATIO EDGE: FLAT
		Group 1 — Hard	
18.71	5676	4766	
20.96	3881	3946	
21.26	4717	3823	
21.42	4335	4417	
21.83	3714	3904	
20.84	4465	4171	1:0.93
		Group 2 — Soft	
22.82	1171	1882	
23.05	2358	2394	
23.37	1706	1838	
23.43	1722	1898	
24.03	1523	1842	
23.34	1696	1971	1:1.16

TABLE XIV — SAMPLE NO. 12

Process of Manufacture..... Semi-Dry Press.
Kind of Clay Used..... Cement and Sand.

ABSORPTION	CRUSHING ON EDGE	CRUSHING ON FLAT	RATIO EDGE: FLAT
15.38	1335	2193	
17.56	998	1544	
19.79	659	1048	
20.31	940	785	
20.71	632	1012	
18.75	913	1316	1:1.44

TABLE XV — SAMPLE NO. 13

Process of Manufacture..... Stiff Mud, Side Cut.
Kind of Clay Used..... Shale.

ABSORPTION	CRUSHING ON EDGE	CRUSHING ON FLAT	RATIO EDGE: FLAT
		Group 1 — Hard	
5.16	9052	7721	
5.42	9713	8400	
5.62	10078	9192	
5.77	10156	8670	
6.27	6148	8165	
5.65	9029	8429	1:0.93
		Group 2 — Medium	
6.36	7892	9175	
6.59	7245	9193	
7.07	11432	8665	
7.22	11023	8175	
7.48	9246	8778	
6.94	9368	8797	1:0.93
		Group 3 — Soft	
7.40	8308	8881	
7.74	9625	7786	
7.78	9675	8115	
7.84	9942	8480	
8.12	7527	8692	
7.78	9015	8391	1:0.93

ANALYSIS OF THE CRUSHING TESTS

The purpose of this portion of this investigation was to get, as accurately as possible, the ratio between the crushing strength on the flat and on the edge, with the idea of possibly being able to translate tests which had been made on one system into roughly approximate figures on the other system. Which ever system may be adopted as standard, the thousands of tests made by the other system ought not to be wholly valueless.

Mean. The first figure sought from the above data was the grand mean. This, taken on 170 bricks, in 34 groups of five bricks each, representing 14 different products, (11 different clays, one being made up by two different methods, one sand-lime brick and one cement-sand brick) gives the following:

TABLE XVI

NUMBER OF BRICKS	AVERAGE ABSORPTION PERCENTAGE	AVERAGE CRUSHING STRENGTH ON EDGE, LBS. PER SQ. IN.	AVERAGE CRUSHING STRENGTH ON FLAT, LBS. PER SQ. IN.	MEAN RATIO
170	14.10	5168	6238	1:1.207

Professor A. V. Bleininger, in the Bureau of Standards Laboratory, working upon 176 samples for the Supervising Architect of the Treasury Department (Vol. 12, *Transactions American Ceramic Society,* page 568) and using the same plan of cutting each brick into two as nearly equal portions as possible and testing one on the flat and one on the edge, obtains very similar results, viz.:

176	....	5399	6226	1:1.153

His samples were all obtained within a radius of 150 miles of Washington, D. C. The instructions were to represent materials of superior quality only. They were therefore, of the harder grades, and practically all made by the stiff-mud process.

Both clay and shale products were represented. The mean absorption percentage of these 176 brick was not given in the tables, but calculated from the data given, was found to be 9.15 percent.

On the assumption that the two series ought to show a closer concordance, if the very soft material in the new series were not considered, so as to confine the comparison to material of about the same average hardness, those results of which the absorption exceeded 17.50 percent were eliminated, and the average absorption and the average crushing strength on edge and on flat were re-calculated. The results show:

TABLE XVII

NUMBER OF BRICKS REPRESENTED	AVERAGE ABSORPTION PERCENTAGE	AVERAGE CRUSHING ON EDGE	AVERAGE CRUSHING ON FLAT	RATIO
100	9.37	7202	8452	1:1.184

A closer study of the relationship between the hardness and this crushing strength ratio as shown later, does not bear out this assumption, and the result shown above is probably fortuitous. In any case, the chief point to be made in this connection is the fairly good agreement between the ratios found by two separate series of tests.

Stiff Mud vs. Soft Mud. Separating the bricks on the line of the process of manufacture, the following comparison is obtained.

TABLE XVIII

	BRICKS	ABSORPTION	CRUSHED ON EDGE	CRUSHED ON FLAT	RATIO
Stiff Mud Process					
Mean	105	10.53	6875	8410	1:1.223
Maximum	5	21.42	1905	4052	1:2.13
Minimum	5	1.19	15139	13232	1:0.87
Soft Mud Process					
Mean	105	10.53	6873	8410	1:1.223
Maximum	5	23.15	1126	1640	1:1.46
Minimum	5	20.84	4465	4171	1:0.93

So far as this data may be taken to indicate a principle, it is that stiff mud bricks, by reason of their more laminated structure, show a higher ratio between crushing on the edge and on the flat, than soft mud bricks do. In this connection, the Bleininger data being practically all from stiff mud bricks, is interesting.

	BRICKS	ABSORPTION	CRUSHED ON EDGE	CRUSHED ON FLAT	RATIO
Mean	176	....	5399	6226	1:1.153

This falls between our stiff-mud mean and our soft-mud mean.

Shale vs. Clay. It is conceivable that the plasticity and cohesive qualities of these two materials may differ so fundamentally that they may make a characteristic structural difference which would be observable in their breaking under pressure. The data was now separated on this line, and is shown in Table XIX.

TABLE XIX

KIND OF CLAY	NUMBER OF BRICKS	AVERAGE PERCENTAGE ABSORPTION	AVERAGE CRUSHING ON EDGE	AVERAGE CRUSHING ON FLAT	RATIOS
Shale	75	7.70	8264	9676	Mean 1.17 Max. 1.75 Min. 0.87
Plastic Clay.....	85	20.50	3022	3893	Mean 1.25 Max. 2.13 Min. 0.94

No principle of importance seems clearly brought out by this comparison.

Hardness of Burning. Assuming that the completeness to which the porosity of the clay is extinguished is a fair measure of its hardness, and dividing the bricks up into groups on the basis of their absorption property alone, and neglecting all considerations of process of manufacture, kind of clay, etc., we get the following:

TABLE XX — VITRIFIED BRICK GROUP

Absorption average not above 5 percent
(Individual tests not exceeding 6 percent)

ABSORPTION	CRUSHED ON EDGE	CRUSHED ON FLAT	RATIOS
percent			
2.06	7659	13262	Max. 1:1.73
3.42	11283	13001	
2.63	10119	13175	
1.19	15139	13232	Min. 1:0.87
2.32	11050	13167	Mean 1:1.19

TABLE XXI — HARD BUILDING BRICK GROUP

Absorption average between 5 and 12 percent
(Individual tests not exceeding 15 percent)

ABSORPTION	CRUSHED ON EDGE	CRUSHED ON FLAT	RATIOS
percent			
5.26	8945	12298	
8.01	5934	10134	Max. 1:1.70
6.05	9790	11932	
6.75	10878	12260	
5.65	9029	8429	
6.94	9368	8797	
7.78	9015	8391	Min. 1:0.93
6.63	8994	10320	Mean 1:1.14

TABLE XXII — COMMON BUILDING BRICK GROUP

Absorption average between 12 and 20 percent
(Individual test not exceeding 24 percent)

ABSORPTION	CRUSHED ON EDGE	CRUSHED ON FLAT	RATIOS
percent			
13.19	4693	6534	
17.67	2580	3088	
19.27	2880	3214	
12.17	5027	5860	
16.67	2835	3996	
16.16	4991	7947	
18.39	4111	7742	Max. 1:1.88
13.95	4876	5742	
16.11	3862	4359	
17.50	3034	2878	Min. 1:0.94
12.75	5659	6253	
12.66	1912	2572	
18.22	2168	2198	
18.75	913	1316	
15.96	3540	4535	Mean 1:1.28

TABLE XXIII — SOFT BRICK GROUP

Absorption average above 20 percent
(Individual tests above 24 percent)

ABSORPTION	CRUSHED ON EDGE	CRUSHED ON FLAT	RATIOS
percent			
23.15	1126	1640	
20.83	1508	2075	
21.42	1905	4052	Max. 1:2.13
21.53	3337	3603	
26.67	1679	2258	
21.43	1421	1937	
20.60	1889	1992	
20.84	4465	4171	Min. 1:0.93
23.34	1696	1971	
22.20	2114	2633	Mean 1:1.24

Assembling the averages of these four groups we have Table XXIV:

TABLE XXIV

GROUP DESIGNATIONS	MEAN ABSORPTION	MEAN CRUSHING ON EDGE	MEAN CRUSHING ON FLAT	MEAN RATIOS
Vitrified Brick	2.32	11050	13167	1:1.19
Hard Brick	6.63	8994	10320	1:1.14
Medium Brick	15.96	3540	4535	1:1.28
Soft Brick	22.20	2114	2633	1:1.24

Table XXIV shows very nicely the intimate relation between the hardness, as represented by the percentage of absorption, and the crushing strength, however it may be measured. This relationship has been fully brought out elsewhere, notably by Bleininger, to which reference has been made. But, no particular light is shed on the question of the ratio between the two modes of making the crushing test. The order of the figures does not suggest any causal relation between hardness and mode of crushing.

Translation of Terms by Calculation. The weight of this evidence seems to establish pretty clearly that the crushing strength developed by bricks in general upon edge is about

83 percent of that when crushed upon the flat, or that the crushing strength upon the edge being taken at 1, the strength upon flat will be about 1.20. Including the evidence brought out by Bleininger, the figures would be 85 percent, or 1 :1.175. But it is quite a different thing to say that in any given case, the strength in one position being known, the strength in the other position could be safely computed. To make such computations of any value, it would have to be shown that not only is the mean value of this factor securely established, but also that the fluctuation of the individual components from the mean is not large.

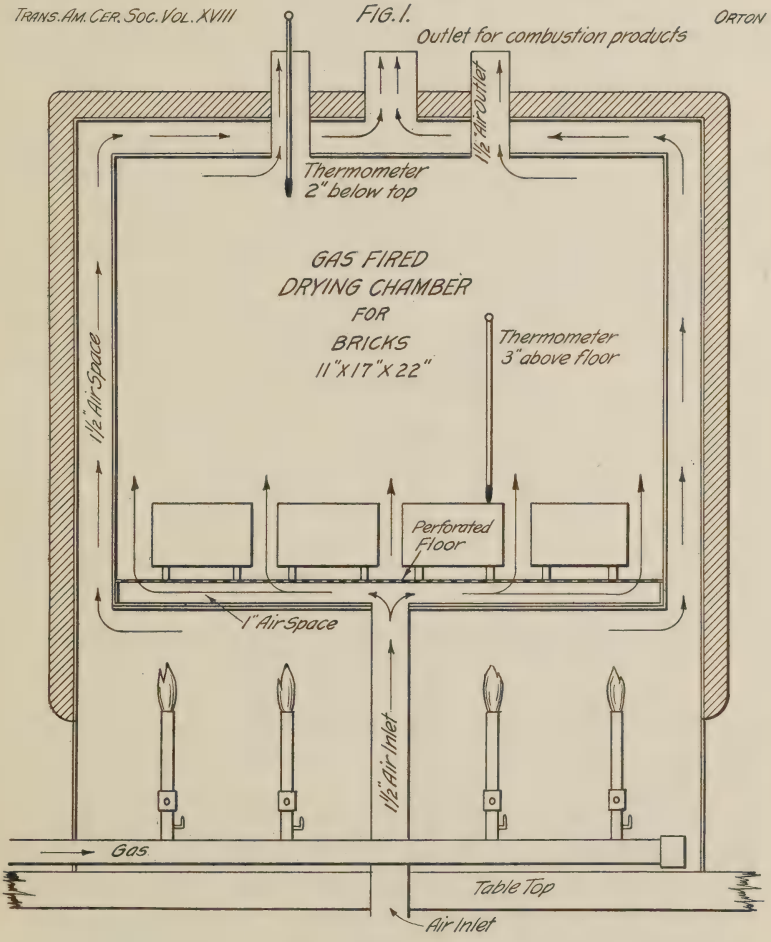
A study of all data on this point shows extreme variability. The mean in this series being 1 : 1.207, the maximum was 1 : 2.13 and the minimum 1 : 0.87. Furthermore, it has thus far been impossible to correlate these wide divergences with any single factor, at least not to any strong degree. It does not seem to be a function of the degree of vitrification, for we find high ratios and low ones in every absorption group. It does not seem to be a function of process of manufacture, for though the most extreme variations in ratios are found among the stiff-mud bricks, the fluctuations in the soft mud group are also wide. It does not seem to be a function of the kind of clay used, for wide ranges of ratio occur in both the shale and clay groups.

It must be further borne in mind that in every instance, we are dealing here with averages of five bricks. If our study descended to comparisons of individual bricks, the fluctuations would undoubtedly go to still wider limits.

So far, then, as the present study shows, we cannot safely translate data made by crushing on the flat into terms of crushing on the edge, except in large masses or averages, and here with many reservations. The fact that crushing on the edge is now coming to be accepted as being the better method will probably lead to the increasing accumulation of data by that process, but for some time yet the store of records of crushing tests made on the flat, will continue to be the ones most generally quoted. All new tests should certainly be done by the *A. S. T. M.* specifications, *i. e.,* half-brick crushed on the edge.

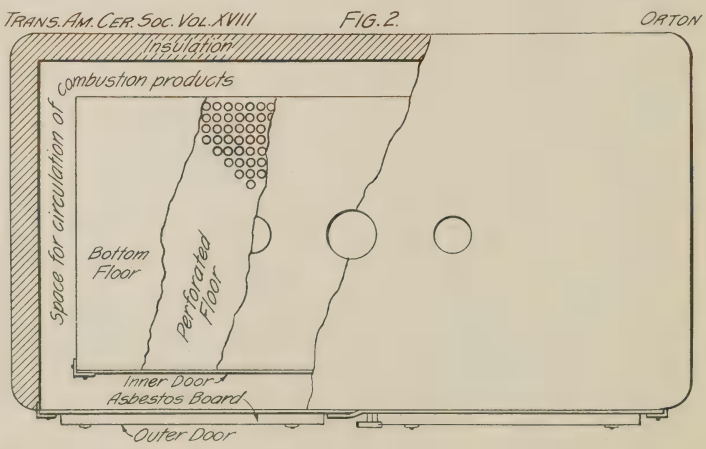
THE FREEZING TEST BY SODIUM SULPHATE

The apparatus and equipment required for this test is exceedingly simple. Beyond the few tanks for soaking the bricks in the sodium sulphate brine, and one for steaming the bricks



before putting into the drier, there is practically nothing required but the driers. Upon these centers the efficiency of the process, for it is rapidity of drying that means rapidity of crystallization, and hence the activity of the process.

The Driers. The drying ovens in which the crystallization takes place required some considerable adjustment. Difficulties arose in securing 110° C. promptly in any small oven, when filled with a charge of cold, saturated brick. The time required to heat the oven is likely to be too long, or the heat distribution is likely to be irregular. A large oven, containing so great a mass of hot material that the introduction of a few charges of wet bricks is unable to chill it off appreciably, is undoubtedly the most desirable tool, but to install and operate such an oven for this test alone is expensive, and would prejudice the wide use of the process. Accordingly our efforts were



directed towards developing a small cheap oven that would do the work fairly well.

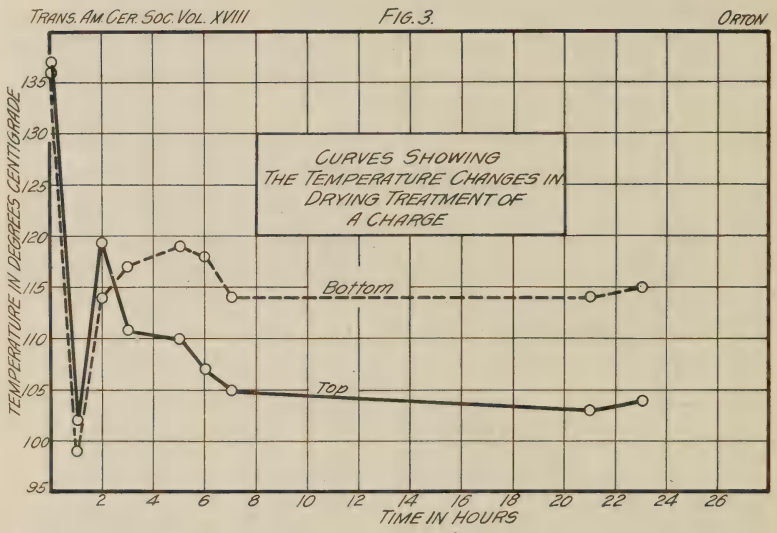
Figures 1 and 2, show the oven that was finally adopted. It was built of sheet iron, with riveted seams. No solder was used. It consists of an inner oven, holding twenty bricks per charge, excluding four on the bottom which are permanent and serve to store and distribute the heat. This inner chamber is ventilated by an inch and one-half inlet pipe bringing in free air, and two and one-half inch outlets from the top. It opens with a pair of close-fitting, over-lapping, inner doors on the front side. It is provided with a sheet-iron, perforated, false floor,

one inch above the real bottom, which distributes the in-coming air supply over the whole cross section.

It is surrounded by an outer sheet iron oven, and separated from it on all sides by a one and one-half inch air space. The outer oven is provided with an inch coating of magnesia-asbestos insulating cement. The heat is supplied by a row of four bunsen burners, connected direct to a one-half inch gas line, and the flames impinge upon perforated discs, riveted to the floor of the inside oven. These discs save the oven floor from burning out, and help to distribute the heat. The hot products of combustion completely surround the inner oven, and escape from a two inch chimney in the center of the top. No combustion gases pass into the inner oven, except by leakage, and as air flows freely in at the bottom and out at the top, there is no difference in pressure to cause any important leakage inwards from the outer air space.

The performance of this oven was found unsatisfactory in the beginning, due to the chilling down from a freshly inserted charge. Even when the oven itself was heated much too hot, the surplus heat stored in it did not go far towards heating up the charge of wet bricks, nor did decreasing the charge and partly filling the oven with preheated dry bricks, remedy this trouble; besides, this seriously cut down the drier's capacity. Finally, the expedient of heating the saturated bricks before placing them in the oven was tried. The bricks were set on supports in a covered tank, containing an inch or so of sulphate brine, and the brine boiled. The bricks were thus surrounded by steam and rapidly took on the steam temperature. When hot through and through, they were quickly transferred to the already heated drier, the door closed, and the operation began.

The following table shows the temperature behavior of the oven after its irregularities had been controlled to the greatest practical point. The measurements were made near the top and bottom of the inner oven.


TRANS. AM CER. SOC. VOL. XVIII
FIG. 3.
ORTON
CURVES SHOWING THE TEMPERATURE CHANGES IN DRYING TREATMENT OF A CHARGE
Bottom
Top
TEMPERATURE IN DEGREES CENTIGRADE
TIME IN HOURS

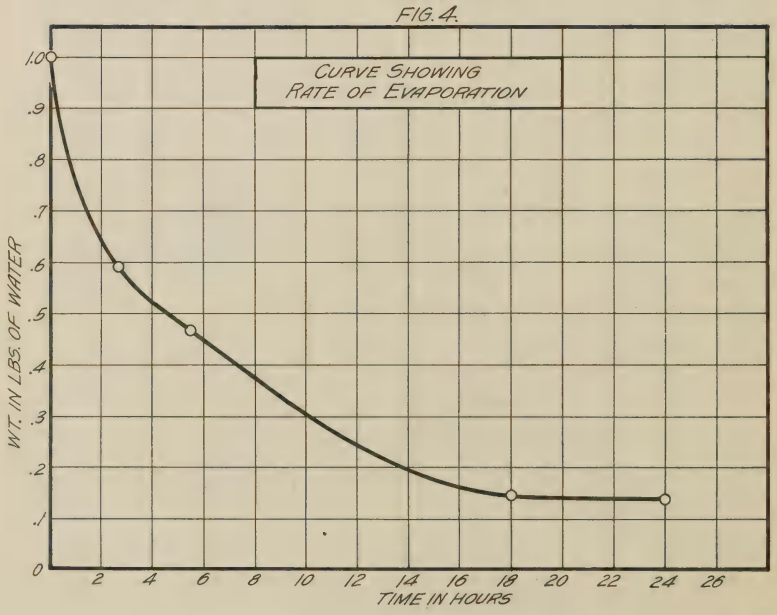


FIG. 4.
CURVE SHOWING RATE OF EVAPORATION
WT. IN LBS. OF WATER
TIME IN HOURS

TABLE XXV

	TOP	BOTTOM	MEAN
Oven pre-heated for reception of charge	137° C.	136° C.	136° C.
One hour after charging	102°	98°	100°
Two hours	120°	114°	117°
Three hours	111°	117°	114°
Five hours	110°	118°	114°
Six hours	107°	118°	112.5°
Seven hours	105°	114°	109.5°
Twenty-one hours	103°	114°	108.5°
Twenty-three hours	104°	115°	109.5°

Figure 3 shows this data in graphic form. It was always impossible to heat the top to 110° without considerably overheating the bottom. The top outstrips the bottom for an hour or two in the beginning, but when the chill caused by the rapid evaporation is over, the bottom passes the top in temperature, being nearer the actual flame.

The apparatus is by no means perfect, and takes attention once an hour in regulating the gas during the first eight hours of a run. Any small oven, no matter how heated or controlled, would have the same general difficulties to contend with, and large ovens are impractical, except in large permanent laboratories where the bulk of work is great. The type of oven here described is cheap, simple, and fairly efficient, if watched with proper care. It costs less than thirty dollars installed, ready for use.

Figure 4 shows the rate at which this drier removed water from a brick under treatment. About 45 percent was expelled in the first three hours, and 55 percent in six hours. The drying was practically complete in eighteen hours.

Figure 5 shows the installation, consisting of three driers used in these tests. This view, supplementing Figures 1 and 2, should enable any one to have a similar oven built by any good tinner.

The Method of Using the Sodium Sulphate Process. The method used was essentially that of the Howat paper, advanced

copy of which was obtained for consultation in this respect. The procedure is as follows:

The dried specimens are immersed in a saturated solution of sodium sulphate at room temperature for twenty-four hours. They were then transferred to a drying oven maintained at 110° C. for twenty-four hours. From the drier, the bricks were again immersed in the sulphate solution for twenty-four hours. To record the effects of each "freeze," the bricks were photographed. It was found that the destructive effects of the "freeze"

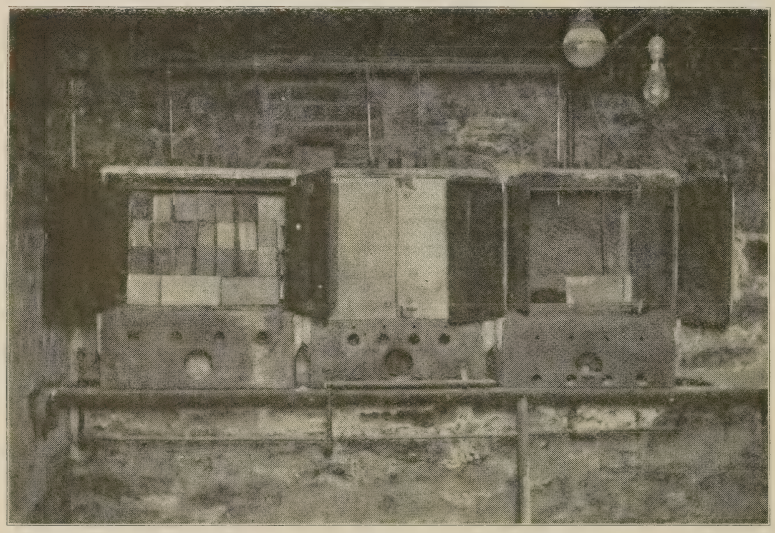


FIG. 5.

were not observed on first taking the brick from the oven, but developed only upon "thawing" in the solution. Accordingly, the photographic records were made after the bricks had been in soak for an hour or more.

The Strength of the Solution. Mr. Howat complained that the process was too severe as compared with the freezing process, and Professor Bleininger corroborated that view. It seemed worth while, therefore, to experiment a little on modes of reducing the severity of the process, before applying it to our extensive series of bricks.

In theory, the destructive action of the sodium sulphate is due to the expansive force of the crystals forming in the pore system of the body treated. If it were possible to completely fill the pores with an anhydrous solution, like that of a fused metal for instance, which would suffer no loss in weight in cooling, but would expand in solidifying, it is evident that the destructive power would be immense, and that nothing could stand it, unless the cohesive strength of the mass exceeded the expansive force due to crystallization.

But with a water solution, of which three-fourths must evaporate before the contained salt has all solidified, it is evident that the bursting pressure cannot be applied with any approach to completeness, for the spaces vacated by the water are much greater than the salt can fill. Hence, disruption of the stone only occurs where a crystal forms in some relatively narrow canal or pore, blocking it in both directions. For this reason, whether it be water or a solution of a salt the first freezing seldom produces very serious disintegration.

In the case of sodium sulphate, or any other salt in a water solution, the first freezing leaves in the pores of the brick all of the salt which the saturated solution brought in. This is a solid substance and decreases the voids of the brick by just that much. Now, if a second treatment be used, and more saturated solution be introduced into the residual voids, and this in turn be crystallized by the removal of the water by evaporation, the new salt added will be a straight addition to that left from the first evaporation, and its expansive force will be exerted in a closer, more confined space than the first time. Each successive impregnation with solution, and crystallization of its contents, will reduce the void spaces of the brick by the volume of the crystals introduced, and will exert its bursting pressure in a more confined space.

In short, the action is cumulative, if a saturated solution is used. The pores become more nearly full of salt with each evaporation, since the incoming solution, being saturated, cannot dissolve any of the salt already there, and adds its own burden on evaporation.

It seemed to the writer that if the first charge of solution were saturated, but that in thawing after the first evaporation, a short treatment in pure water were used, the sulphate crystals in the pores of brick would be re-dissolved, and therefore re-crystallization could only apply once more the same expansive force exerted by the crystals carried in on the first charge. Similar repetitions of the crystallization and solution, would be dealing with the same expansive force each time, and the pore system would not become choked, and unable to give relief to the pressure. Such a process should certainly slow down the destructive power complained of in the Howat paper.

Accordingly, three batches of bricks not needed in the principal investigation were prepared and submitted to the two treatments, side by side. In the one, the solution used was saturated; in the other, the first immersion was in saturated solution, but the thawing was done in rain water for one hour, and the bricks were then transferred to saturated brine for the twenty or so hours remaining, prior to placing again in the drier. The purpose of this one hour treatment in pure water was to permit re-solution of the first charge of crystals each time, and to fill the voids of the brick with this solution. The soaking in the saturated brine afterwards, was to safeguard loss of the initial charge of sodium sulphate from the brick, by leaching.

The results of these preliminary tests are shown in Table XXVI.

TABLE XXVI — SAMPLE NO. 3

Process of Manufacture..... Soft Mud.
Kind of Clay..... Glacial.

	SATURATED SOLUTION TREATMENT		MODIFIED TREATMENT	
Absorption percents of bricks	Brick 1	22.62	Brick 6	21.68
	2	20.20	7	20.59
	3	19.68	8	20.08
	4	19.60	9	19.65
	5	19.47	10	19.56
	Average	20.31	Average	20.31
First treatment	No noticeable effects		No noticeable effects	
Second treatment	No. 1 considerably affected No. 4 slightly affected		No noticeable effects	
Third Treatment	No. 1 badly shelled off No. 3 corner off		No noticeable effects	
Fourth treatment	No. 1 gone No. 3 corner more destroyed No. 5 corner begun to go		No marked effects	
Fifth treatment	No. 1 gone No. 3 ends gone No. 4 edge gone No. 5 end gone		Slight effects noticed for first time	
Sixth treatment	Effects slightly increased on all remaining bricks. No. 5 almost gone		Not much change	
Seventh treatment	Not continued		Edges begun chipping off from 6-7-8 and 10	
Eighth treatment	Not continued		No increased effect apparent	
Ninth Treatment	Not continued		Edges and ends of No. 9 begin chipping off	
Tenth treatment	Not continued		Very slight change	
Eleventh treatment	Not continued		Very slight change	
Twelfth treatment	Not continued		Very slight change	

TABLE XXVII — SAMPLE NO. 7

Process of Manufacture..... Soft Mud.
Kind of Clay..... Glacial.

	SATURATED SOLUTION TREATMENT		MODIFIED TREATMENT	
Absorption of test pieces	Brick 1	18.41	Brick 6	18.84
	2	20.79	7	20.91
	3	20.87	8	20.82
	4	20.87	9	20.68
	5	20.44	10	20.14
	Average	20.27	Average	20.27
First treatment	No noticeable effect		No noticeable effect	
Second treatment	No noticeable effect		No noticeable effect	
Third treatment	No. 2 begins to fail		No noticeable effect	
Fourth treatment	No. 1 and No. 2 gone, 3, 4 and 5 affected seriously		No marked effect.	
Fifth treatment	Nos. 3, 4 and 5 still holding together but badly flaked		Slight general effect can be noticed	
Sixth treatment	Nos. 3, 4, 5 still remaining, but badly defaced		Slight general effect can be noticed	
Seventh treatment	Not continued after sixth treatment		Noticeable increase in attack. All superficial	
Twelfth treatment			All bricks survive, but Nos. 8 and 9 pretty badly defaced. All somewhat affected	

TABLE XXVIII — SAMPLE NO. 10

Process of Manufacture..... Soft Mud.
Kind of Clay..... Glacial.

	SATURATED SOLUTION TREATMENT		MODIFIED TREATMENT	
Absorption of test pieces	Brick 1	19.65	Brick 6	19.56
	2	19.80	7	19.77
	3	19.29	8	19.96
	4	20.17	9	20.17
	5	20.13	10	20.08
	Average	19.81	Average	19.91
First treatment	No noticeable effect		No noticeable effect	
Second treatment	No 1 starts to crumble No. 3 seriously attacked		No noticeable effect	
Third treatment	No. 4 one end affected others all in bad shape		Slight noticeable effect	
Fourth treatment	No. 4 one end off, 1-2-3-5 gone		Slight noticeable effect	
Fifth treatment	No. 4 still holding together		Slight disintegration on all samples	
Sixth treatment	No. 4 still holding together		No. 7 loses an end	
Twelfth treatment	Discontinued at sixth treatment		All bricks whole, corners gone, surface somewhat flaked	

In order to enable these impressions to be more fully grasped, Figs. 6 and 7 reproduce the photographs of the two series at the fourth and twelfth treatments respectively.

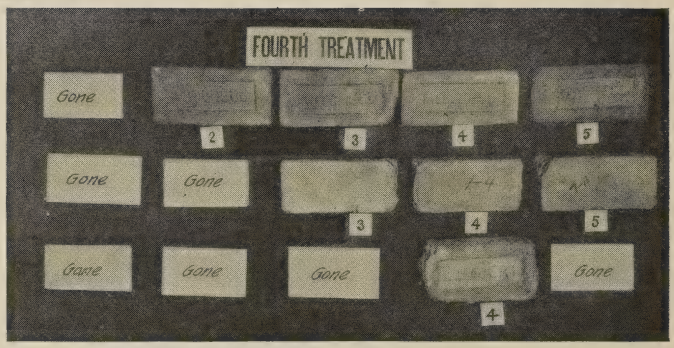
The results of these tests showed several things:

1st. That under the standard process, using only saturated solution at every immersion, all three samples were quite rapidly attacked. All were soft bricks, of rather high absorption.

2nd. Bricks of practically identical absorption, but of different makes, resist the action of the sulphate quite differently.

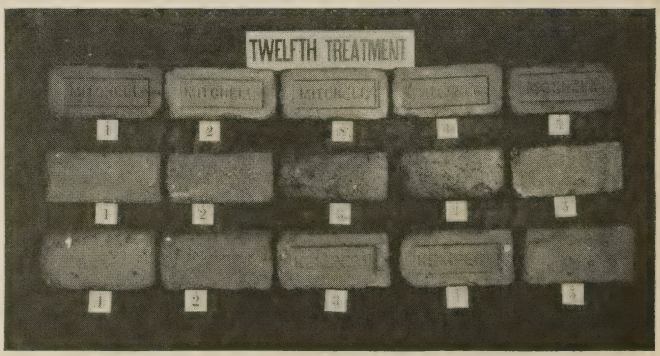
In the fourth treatment, one sample had only one mutilated brick left. Another sample, of slightly higher absorption, had four bricks left, in relatively good condition.

TRANS. AM. CER SOC. VOL. XVIII FIG. 6. ORTON



3rd. By use of pure water at the beginning of each thawing for an hour, the severity of the test is very greatly mitigated. Twelve treatments have not produced nearly so much destruction as four treatments by the standard process.

TRANS. AM. CER SOC. VOL. XVIII FIG. 7. ORTON



4th. The severity of the standard process, while undoubted in the case of very weak material, should not be condemned until tried more fully on a series involving a complete assortment.

On the strength of these experiments with the apparatus and with the method, it was now decided to go ahead upon the material of the principal series.

The Regular Series. This involved giving five repetitions of the treatment with saturated sodium sulphate brine,

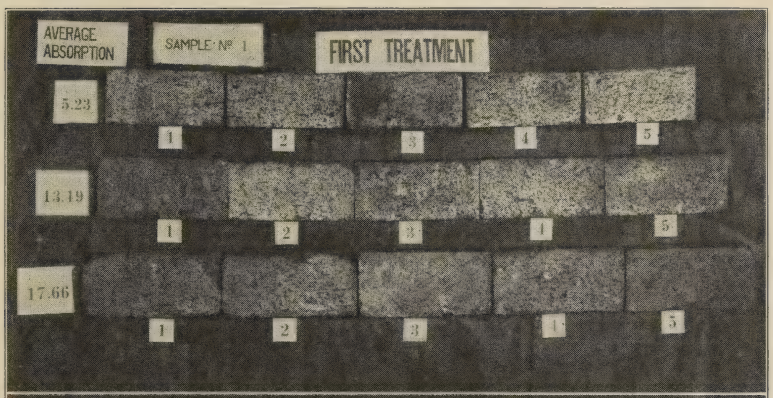


FIG. 8.

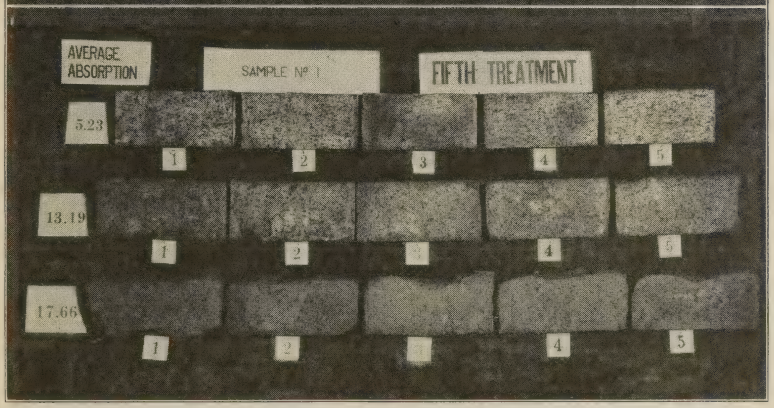


FIG. 9.

making photographs after each complete treatment to enable study of the progressive changes, upon 34 lots of 5 bricks each. Each lot required ten days to come through, but two could be carried forward together, using the drier on alternate days. The whole work with the sulphate took about two months.

In order to condense the data, the following symbols were adopted to express the various stages of destruction which the different samples exhibited:

TABLE XXIX

SYMBOL	INTREPRETATION	NUMBER OF SAMPLE ILLUSTRATING THIS TYPE
A	No apparent effect	Sample 1 Group 1 Figure 9
B	Very slight superficial disintegration, without cracking No apparent loss of strength	Sample 12 Group 1 Figure 31
C	Considerable superficial disintegration—loss of some corners or edges probable or Development of small cracks, without disintegration No evidence of marked internal weakening	Sample 1 Group 3 Figure 9 or Sample 2 Group 1 Figure 11
D	Severe superficial disintegration with loss of corners and edges or Development of serious structural cracks Internal weakening evident	Sample 4 Group 3 Figure 15
E	Mutilation by splitting off of large pieces, or breaking into bats Structure shaky and strength practically gone	Sample 5 Group 3 Figure 17
F	Complete failure	

For convenience, these symbols will be called Disintegration Coefficients.

The tables which follow are arranged to bring similar processes of manufacture together, and without regard to the order of the sample numbers.

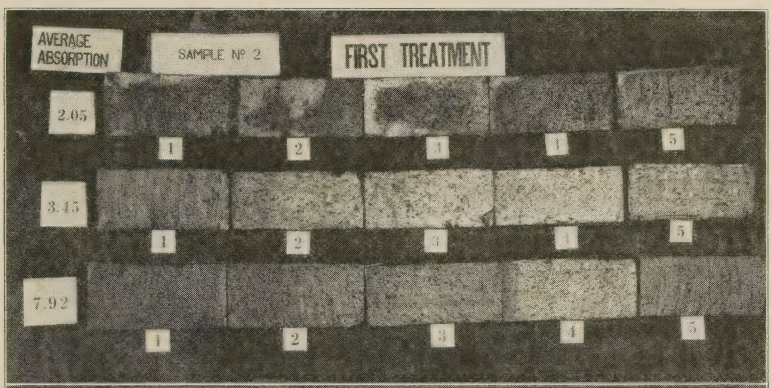

AVERAGE ABSORPTION
SAMPLE № 2
FIRST TREATMENT
2.05
3.45
7.92
1
2
3
4
5

Fig. 11.

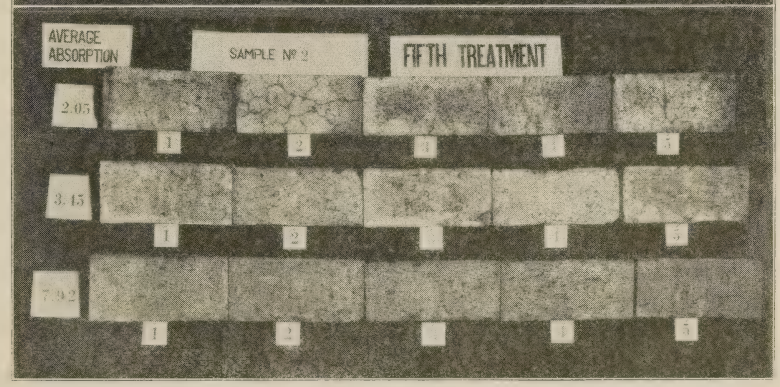

AVERAGE ABSORPTION
SAMPLE № 2
FIFTH TREATMENT
2.05
3.45
7.92
1
2
3
4
5

Fig. 10.

TABLE XXX — SAMPLE NO. 1

Process of Manufacture..... Stiff Mud, Side Cut.
Kind of Clay Used..... Shale.

GROUP NO. AND MFR'S GRADING	MEAN CRUSHING STRENGTH ON EDGE	ABSORPTION DATA		CONDITION OF BRICKS AFTER EACH SULPHATE TREATMENT EXPRESSED IN DISINTEGRATION COEFFICIENTS				
		BRICK NO.	PERCENT	FIRST TREATMENT	SECOND TREATMENT	THIRD TREATMENT	FOURTH TREATMENT	FINAL TREATMENT
1 Hard	8945	1	4.33	A	A	A		A
		2	5.23	A	A	A		A
		3	6.14	A	A	A	No	A
		4	5.32	A	A	A	data	A
		5	5.14	A	A	A		A
		Mean	5.23					
2 Medium	4693	1	12.62	A	A	A		A
		2	12.80	A	A	B		B
		3	13.10	A	A	A	No	A
		4	13.57	A	A	A	data	A
		5	13.88	A	A	B		B
		Mean	13.19					
3 Soft	2580	1	18.77	A	A	B		C
		2	18.64	A	A	B		C
		3	17.86	A	A	B	No	C
		4	17.71	A	A	B	data	C
		5	15.35	A	A	B		C
		Mean	17.66					

REMARKS. This was a well selected series, representing fairly the capacity of this clay, made up by this process. The softest grade, after five treatments, was considerably defaced, but not weakened internally, so far as could be judged.

TABLE XXXI — SAMPLE NO. 2

Process of Manufacture..... Stiff Mud, Side Cut.
Kind of Clay Used..... Shale.

GROUP NO. AND MAKERS' GRADING	MEAN CRUSHING STRENGTH ON EDGE	ABSORPTION DATA		CONDITION OF BRICKS AFTER EACH SULPHATE TREATMENT EXPRESSED IN DISINTEGRATION COEFFICIENTS				
		BRICK NO.	PERCENT	FIRST TREATMENT	SECOND TREATMENT	THIRD TREATMENT	FOURTH TREATMENT	FIFTH TREATMENT
1 Hard	7659	1	1.97	A	A	A	A	A
		2	1.34	A	A	C	C	C
		3	2.21	A	A	A	A	A
		4	2.22	A	A	A	A	A
		5	2.52	A	A	A	A	A
		Mean	2.05					
2 Medium	11283	1	4.38	A	A	A	A	A
		2	3.30	A	A	A	A	A
		3	3.70	A	A	A	A	A
		4	3.10	A	A	A	A	A
		5	2.68	A	A	A	A	A
		Mean	3.45					
3 Soft	5934	1	9.12	A	A	A	A	A
		2	8.97	A	A	A	A	A
		3	8.04	A	A	A	A	A
		4	7.26	A	A	A	A	A
		5	6.40	A	A	A	A	A
		Mean	7.92					

REMARKS. This series was not well selected. The soft set should have been graded medium, and much softer material furnished for the last group.

The one brick which was affected was evidently steam-checked in drying or burning, as the surface was a mass of intersecting cracks, very shallow, and apparently not injuring the strength.

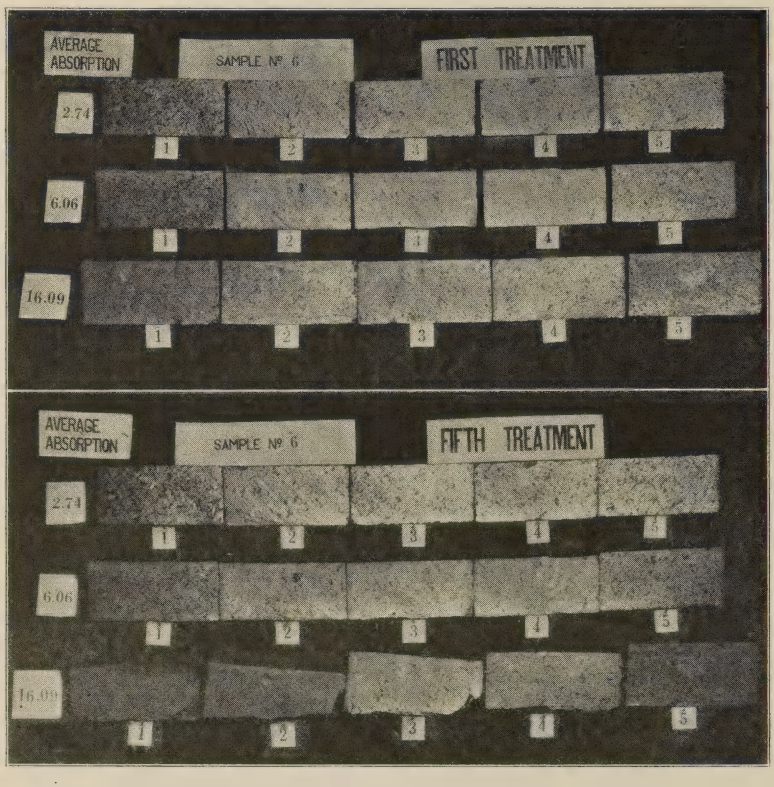

AVERAGE ABSORPTION
SAMPLE Nº 6
FIRST TREATMENT
2.74
6.06
16.09
1
2
3
4
5

FIG. 12.

AVERAGE ABSORPTION
SAMPLE Nº 6
FIFTH TREATMENT
2.74
6.06
16.09
1
2
3
4
5

FIG. 13.

TABLE XXXII — SAMPLE NO. 6

Process of Manufacture..... Stiff Mud, Side Cut.
Kind of Clay Used..... Shale.

GROUP NO. AND MAKERS' GRADING	MEAN CRUSHING STRENGTH ON EDGE	ABSORPTION DATA		CONDITION OF BRICKS AFTER EACH SULPHATE TREATMENT EXPRESSED IN DISINTEGRATION COEFFICIENTS				
		BRICK NO.	PERCENT	FIRST TREATMENT	SECOND TREATMENT	THIRD TREATMENT	FOURTH TREATMENT	FIFTH TREATMENT
1 Hard	10119	1	2.35	A	A	A	A	A
		2	2.00	A	A	A	A	A
		3	1.82	A	A	A	A	A
		4	3.61	A	A	A	A	A
		5	3.94	A	A	A	A	A
		Mean	2.74					
2 Medium	9790	1	4.30	A	A	A	A	A
		2	5.37	A	A	A	A	A
		3	6.00	A	A	A	A	A
		4	7.02	A	A	A	A	A
		5	7.61	A	A	A	A	A
		Mean	6.06					
3 Soft	3862	1	17.35	A	C	D	D	E
		2	16.88	A	A	D	D	E
		3	16.48	A	A	D	D	E
		4	15.54	A	A	C	D	E
		5	14.21	A	A	A	C	E
		Mean	16.09					

REMARKS. A well selected series, with proper extremes.
These bricks break down by cracking along their lamination planes, not by surface disintegration. The third group was ready to go to pieces completely after the fifth treatment.

TABLE XXXIII — SAMPLE NO. 8

Process of Manufacture..... Stiff Mud, Side Cut.
Kind of Clay Used..... Shale.

GROUP NO. AND MAKERS' GRADING	MEAN CRUSHING STRENGTH ON EDGE	ABSORPTION DATA		CONDITION OF BRICKS AFTER EACH SULPHATE TREATMENT EXPRESSED IN DISINTEGRATION COEFFICIENTS				
		BRICK NO.	PERCENT	FIRST TREATMENT	SECOND TREATMENT	THIRD TREATMENT	FOURTH TREATMENT	FIFTH TREATMENT
1 Hard	15139	1	0.80	A	A	A	A	A
		2	1.01	A	A	A	A	A
		3	1.01	A	A	A	A	A
		4	1.35	A	A	A	A	A
		5	1.99	A	A	A	A	A
		Mean	1.22					
2 Medium	10878	1	5.71	A	A	A	A	A
		2	6.60	A	A	A	A	A
		3	7.44	A	A	A	A	A
		4	7.33	A	A	A	A	A
		5	6.96	A	A	A	A	A
		Mean	6.81					
3 Soft	5669	1	13.55	A	A	A	A	A
		2	13.34	A	A	A	A	A
		3	12.39	A	A	A	A	A
		4	12.27	A	A	A	A	A
		5	12.15	A	A	A	A	A
		Mean	12.74					

REMARKS. This was a well selected Series, with extremes both represented.

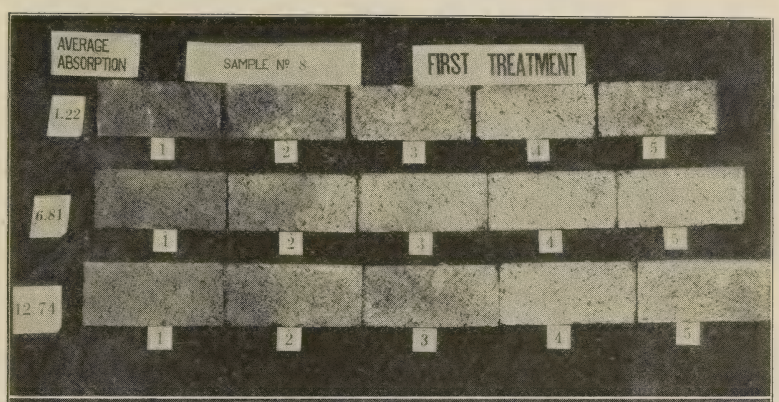

AVERAGE ABSORPTION
SAMPLE Nº 8
FIRST TREATMENT
1.22
6.81
12.74
1
2
3
4
5

FIG. 14.

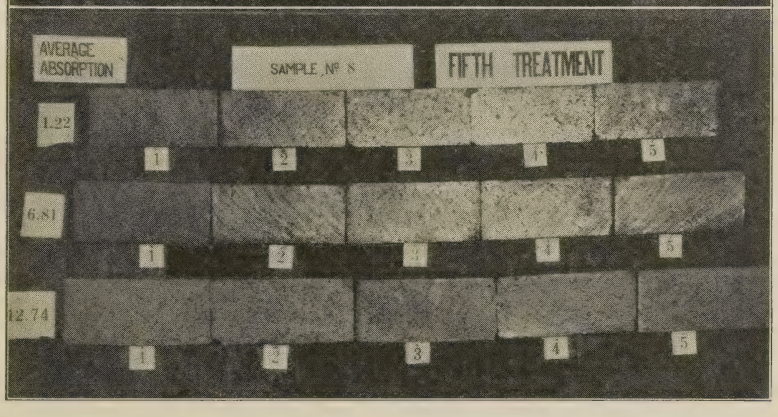

AVERAGE ABSORPTION
SAMPLE Nº 8
FIFTH TREATMENT
1.22
6.81
12.74
1
2
3
4
5

FIG. 15.

TABLE XXXIV — SAMPLE NO. 13

Process of Manufacture..... Stiff Mud, Side Cut.
Kind of Clay Used..... Shale.

GROUP NO. AND MAKERS GRADING	MEAN CRUSHING STRENGTH ON EDGE	ABSORPTION DATA		CONDITION OF BRICKS AFTER EACH SULPHATE TREATMENT EXPRESSED IN DISINTEGRATION COEFFICIENTS				
		BRICK NO.	PERCENT	FIRST TREATMENT	SECOND TREATMENT	THIRD TREATMENT	FOURTH TREATMENT	FIFTH TREATMENT
1 Hard	9029	1	5.04	A	A	A	A	A
		2	5.07	A	A	A	A	A
		3	5.05	A	A	A	A	A
		4	5.53	A	A	A	A	A
		5	5.98	A	A	A	A	A
		Mean	5.33					
2 Soft	9015	1	7.36	A	A	A	A	A
		2	7.36	A	A	A	A	A
		3	7.21	A	A	A	A	A
		4	7.25	A	A	A	A	A
		5	7.00	A	A	A	A	A
		Mean	7.24					
Not furnished								

REMARKS. This series was not well selected. The difference between grades is too little to give an idea of the real limits of the material.

TABLE XXXV — SAMPLE NO. 5

Process of Manufacture..... Stiff Mud, Side Cut.
Kind of Clay Used..... Glacial, Surface Material—Plastic.

GROUP NO. AND MAKERS' GRADING	MEAN CRUSHING STRENGTH ON EDGE	ABSORPTION DATA		CONDITION OF BRICKS AFTER EACH SULPHATE TREATMENT EXPRESSED IN DISINTEGRATION COEFFICIENTS				
		BRICK NO.	PERCENT	FIRST TREATMENT	SECOND TREATMENT	THIRD TREATMENT	FOURTH TREATMENT	FIFTH TREATMENT
1 Hard	4991	1	15.39	A	A	B	B	C
		2	15.66	A	A	A	A	A
		3	15.45	A	A	A	A	A
		4	17.15	A	A	A	A	C
		5	16.93	A	A	A	A	A
		Mean	16.12					
2 Medium	4111	1	18.10	A	A	A	A	A
		2	17.96	A	A	A	A	A
		3	17.59	A	A	A	A	A
		4	20.00	A	A	A	B	C
		5	18.37	A	A	A	B	C
		Mean	18.40					
3 Soft	1905	1	21.05	A	A	D	F	—
		2	21.77	A	A	D	E	E
		3	21.58	A	A	C	D	E
		4	21.48	A	A	C	D	E
		5	21.27	A	A	D	E	E
		Mean	21.43					

REMARKS. This series did not contain a group of sufficiently hard material to make it representative. No. 1 should have been called medium and a new set of 10-12 percent absorption, or less, obtained for the hard group.
This material fails along lamination planes — not by granulation.

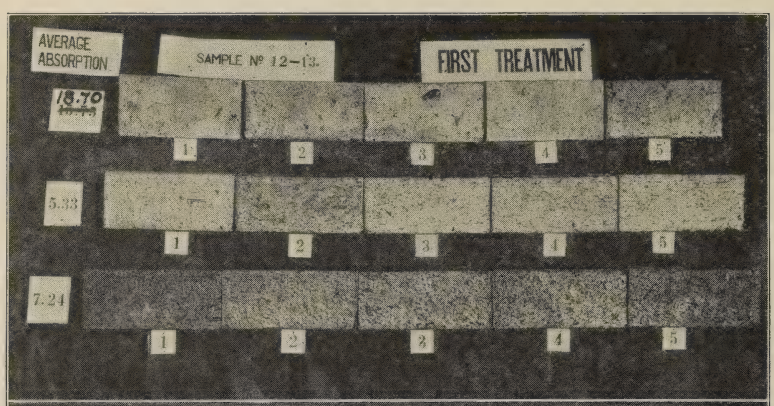

AVERAGE ABSORPTION
SAMPLE Nº 12-13.
FIRST TREATMENT
18.70
1 2 3 4 5
5.33
1 2 3 4 5
7.24
1 2 3 4 5

FIG. 16.

AVERAGE ABSORPTION
SAMPLE Nº 12-13
FIFTH TREATMENT
18.70
1 2 3 4 5
5.33
1 2 3 4 5
7.24
1 2 3 4 5

FIG. 17.

TABLE XXXVI — SAMPLE NO. 4

Process of Manufacture..... Stiff Mud, End Cut.
Kind of Clay Used..... Alluvial, Plastic Material.

GROUP NO. AND MAKERS' GRADING	MEAN CRUSHING STRENGTH ON EDGE	ABSORPTION DATA		CONDITION OF BRICKS AFTER EACH SULPHATE TREATMENT EXPRESSED IN DISINTEGRATION COEFFICIENTS				
		BRICK NO.	PERCENT	FIRST TREATMENT	SECOND TREATMENT	THIRD TREATMENT	FOURTH TREATMENT	FIFTH TREATMENT
1 Hard	5207	1	13.75	A	A	A	A	A
		2	12.60	A	A	A	A	A
		3	12.30	A	A	A	A	A
		4	12.11	A	A	A	A	A
		5	10.41	A	A	A	A	A
		Mean	12.23					
2 Medium	2835	1	15.46	A	A	A	A	A
		2	16.05	A	A	A	A	A
		3	15.65	A	A	B	B	C
		4	18.96	A	A	A	A	A
		5	17.25	A	B	B	B	C
		Mean	16.67					
3 Soft	1508	1	22.39	A	B	C	D	F
		2	21.95	A	C	C	C	D
		3	20.96	A	F	—	—	—
		4	19.70	A	C	D	D	D
		5	19.10	A	C	C	D	D
		Mean	20.82					

REMARKS. A pretty well selected series.

These bricks fail by cracking in flakes along the lamination planes. Being end cuts, their cracks run differently from the others preceding, but proceed from the same cause.

TABLE XXXVII — SAMPLE NO. 3

Process of Manufacture..... Soft Mud Machine.
Kind of Clay..... Plastic Glacial Surface Material.

GROUP NO. AND MAKERS' GRADING	MEAN CRUSHING STRENGTH ON EDGE	ABSORPTION DATA		CONDITION OF BRICKS AFTER EACH SULPHATE TREATMENT EXPRESSED IN DISINTEGRATION COEFFICIENTS				
		BRICK NO.	PERCENT	FIRST TREATMENT	SECOND TREATMENT	THIRD TREATMENT	FOURTH TREATMENT	FIFTH TREATMENT
1 Hard	2880	1	18.89	A	A	A	A	A
		2	18.28	A	A	A	A	A
		3	19.96	A	A	A	A	A
		4	19.80	A	A	A	A	A
		5	19.42	A	A	A	A	A
		Mean	19.27					
2 Soft	1126	1	26.26	C	D	F	—	—
		2	24.50	C	D	F	—	—
		3	23.43	C	E	F	—	—
		4	22.35	A	A	A	A	A
		5	20.28	A	A	A	A	A
		Mean	23.36					
3 Not furnished								

REMARKS. This series was probably well selected. The difference in mean absorption is not great, but the line between safe burning and too soft burning is clearly marked. Group two especially emphasizes this. These bricks disintegrate, rather split.

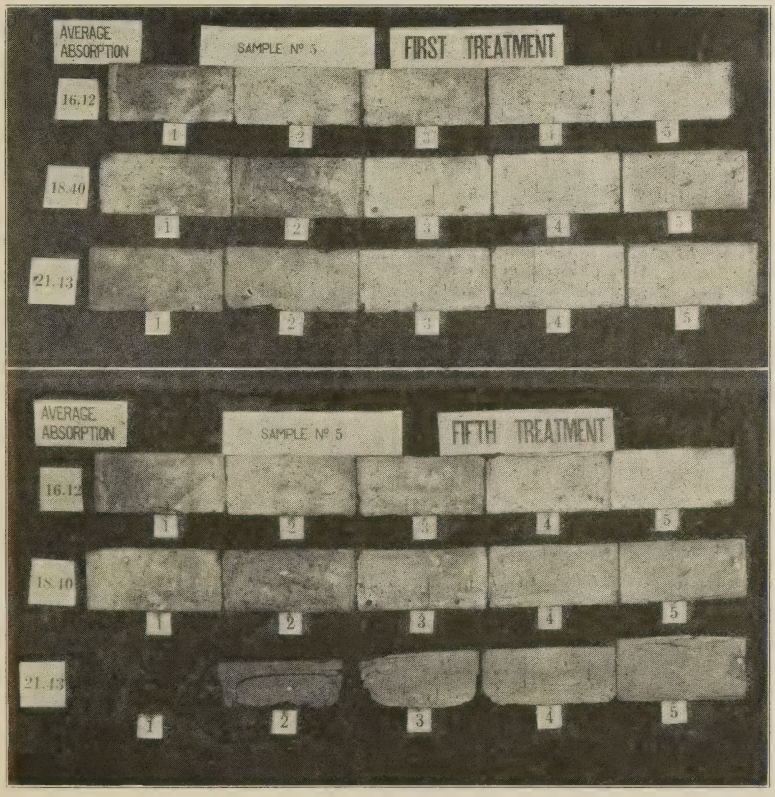

AVERAGE ABSORPTION
SAMPLE Nº 5
FIRST TREATMENT
16.12
18.40
21.13
1
2
3
4
5

FIG. 18.

AVERAGE ABSORPTION
SAMPLE Nº 5
FIFTH TREATMENT
16.12
18.40
21.13
1
2
3
4
5

FIG. 19.

TABLE XXXVIII — SAMPLE NO. 5-A

Process of Manufacture..... Soft Mud Machine.
Kind of Clay Used..... Plastic Glacial Material.

GROUP NO. AND MAKERS' GRADING	MEAN CRUSHING STRENGTH ON EDGE	ABSORPTION DATA		CONDITION OF BRICKS AFTER EACH SULPHATE TREATMENT EXPRESSED IN DISINTEGRATION COEFFICIENTS				
		BRICK NO.	PERCENT	FIRST TREATMENT	SECOND TREATMENT	THIRD TREATMENT	FOURTH TREATMENT	FIFTH TREATMENT
1 Hard	4876	1	12.14	A	A	A	A	A
		2	12.94	A	A	A	A	A
		3	12.61	A	A	A	A	A
		4	14.52	A	A	A	A	A
		5	17.39	A	A	A	A	A
		Mean	13.92					
2 Medium	3337	1	19.74	A	A	A	C	C
		2	20.10	A	A	A	A	A
		3	21.24	A	A	A	A	A
		4	23.19	A	A	C	C	C
		5	23.41	A	A	A	A	A
		Mean	21.53					
3 Soft	1679	1	24.78	A	A	A	A	A
		2	29.60	C	E	F	—	—
		3	28.31	A	E	F	—	—
		4	26.41	A	A	A	A	A
		5	25.21	A	A	A	A	A
		Mean	26.86					

REMARKS. This was a well selected series. The clay will not burn to a dense body, at any temperature.

The test has picked out the two soft burned samples that are clearly over the line, very promptly.

TABLE XXXIX — SAMPLE NO. 7

Process of Manufacture..... Soft Mud Machine.
Kind of Clay Used..... Plastic Glacial Material.

GROUP NO. AND MAKERS' GRADING	MEAN CRUSHING STRENGTH ON EDGE	ABSORPTION DATA		CONDITION OF BRICKS AFTER EACH SULPHATE TREATMENT EXPRESSED IN DISINTEGRATION COEFFICIENTS				
		BRICK NO.	PERCENT	FIRST TREATMENT	SECOND TREATMENT	THIRD TREATMENT	FOURTH TREATMENT	FIFTH TREATMENT
1 Hard	3034	1	16.30	A	A	C	C	C
		2	17.83	A	A	D	D	E
		3	17.80	A	A	D	D	E
		4	17.48	A	C	C	C	D
		5	18.09	A	B	B	B	C
		Mean	17.50					
2 Soft	1421	1	21.92	A	C	C	D	E
		2	21.42	A	C	C	D	E
		3	21.32	A	F	—	—	—
		4	21.19	A	F	—	—	—
		5	21.23	A	F	—	—	—
		Mean	21.41					
Not furnished								

REMARKS. This clay apparently does not develop a perfectly safe structure at the absorption 17.50. It was very poorly prepared, with big pebbles, etc., in it and this probably made even fairly strong hard bricks vulnerable to the sulphate attack.

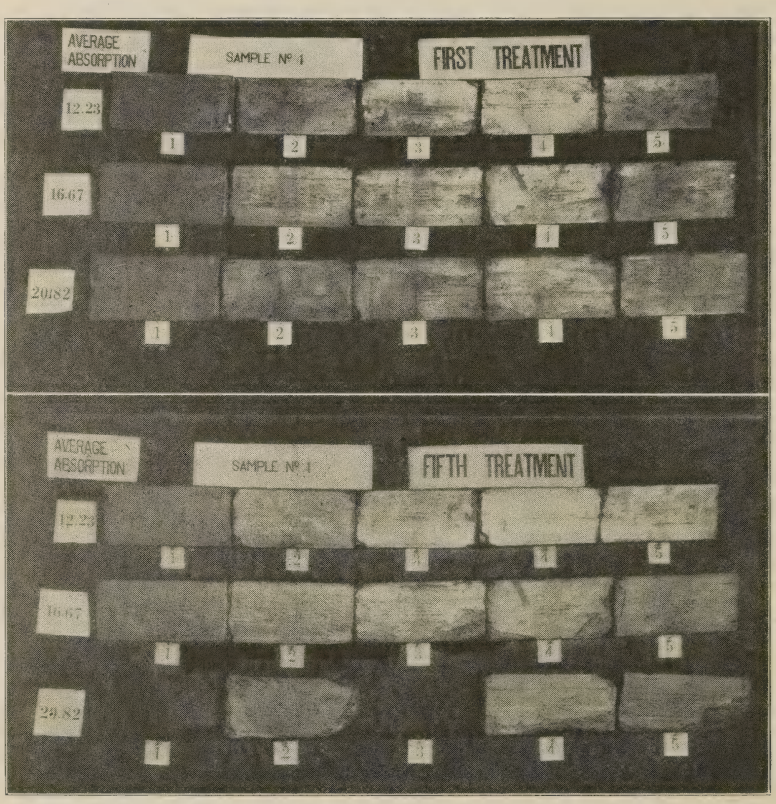

AVERAGE ABSORPTION
SAMPLE Nº 4
FIRST TREATMENT
12.23
16.67
20.82
1
2
3
4
5

FIG. 20.

AVERAGE ABSORPTION
SAMPLE Nº 4
FIFTH TREATMENT
12.23
16.67
20.82
1
2
3
4
5

FIG. 21.

TABLE XL — SAMPLE NO. 10

Process of Manufacture..... Soft Mud Machine.
Kind of Clay Used..... Plastic Glacial Material.

GROUP NO. AND MAKERS' GRADING	MEAN CRUSHING STRENGTH ON EDGE	ABSORPTION DATA		CONDITION OF BRICKS AFTER EACH SULPHATE TREATMENT EXPRESSED IN DISINTEGRATION COEFFICIENTS				
		BRICK NO.	PERCENT	FIRST TREATMENT	SECOND TREATMENT	THIRD TREATMENT	FOURTH TREATMENT	FIFTH TREATMENT
1 Hard	2168	1	17.98	A	B	B	C	C
		2	17.65	A	B	B	B	B
		3	18.45	A	F	—	—	—
		4	19.15	A	F	—	—	—
		5	18.47	A	F	—	—	—
		Mean	18.34					
2 Soft	1889	1	20.84	A	F	—	—	—
		2	20.88	A	B	C	D	E
		3	20.74	A	B	C	D	E
		4	20.30	A	C	D	D	E
		5	20.21	A	D	F	—	—
		Mean	20.59					
Not furnished								

REMARKS. This series is not widely enough differentiated to show sharp differences between what the maker has graded as Hard and Soft. The two best preserved bricks are the two lowest in absorption.

TABLE XLI — SAMPLE NO. 11

Process of Manufacture..... Soft Mud Machine.
Kind of Clay Used..... Plastic Glacial Material.

GROUP NO. AND MAKERS' GRADING	MEAN CRUSHING STRENGTH ON EDGE	ABSORPTION DATA		CONDITION OF BRICKS AFTER EACH SULPHATE TREATMENT EXPRESSED IN DISINTEGRATION COEFFICIENTS				
		BRICK NO.	PERCENT	FIRST TREATMENT	SECOND TREATMENT	THIRD TREATMENT	FOURTH TREATMENT	FIFTH TREATMENT
1 Hard	4465	1	18.48	A	A	A	A	A
		2	21.00	A	A	A	A	A
		3	21.37	A	A	A	A	A
		4	21.51	A	A	A	A	A
		5	21.55	A	A	A	A	A
		Mean	20.78					
2 Soft	1696	1	23.88	A	B	C	D	E
		2	23.81	A	B	C	D	E
		3	23.26	A	B	F	—	—
		4	22.94	A	A	B	D	E
		5	22.79	A	A	C	D	E
		Mean	23.34					
Not furnished								

REMARKS. These samples were not widely differentiated in absorption, but the process drew the line between the fit and unfit astonishingly sharply.

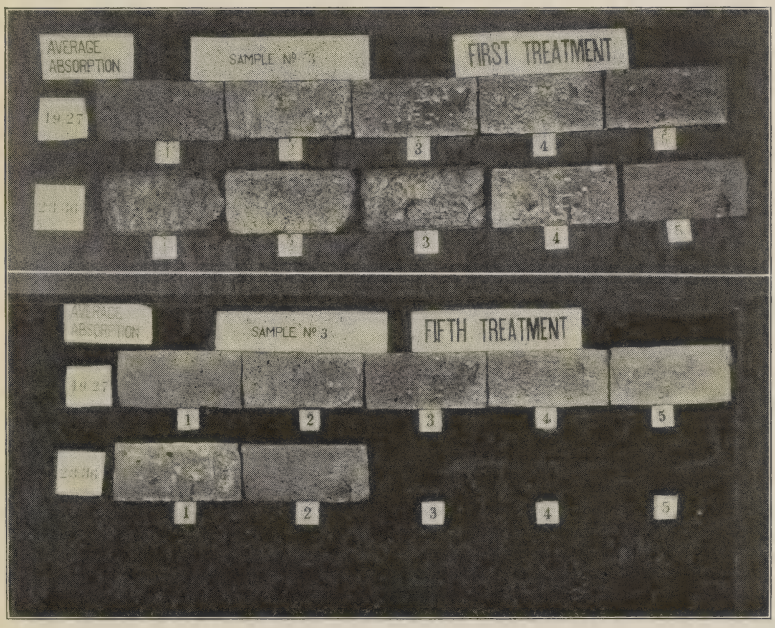

AVERAGE ABSORPTION
SAMPLE Nº 3
FIRST TREATMENT
1927
1
2
3
4
5
1
2
3
4
5

FIG. 22.

SAMPLE Nº 3
FIFTH TREATMENT
1927
1
2
3
4
5
1
2
3
4
5

FIG. 23.

TABLE XLII — SAMPLES NO. 9 AND 12

Process of Manufacture..... Semi-dry Press, and Hydraulic Bond.
Materials ..... Sand with Lime or Cement.

VARIETY	MEAN CRUSHING STRENGTH ON EDGE	ABSORPTION DATA		CONDITION OF BRICKS AFTER EACH SULPHATE TREATMENT EXPRESSED IN DISINTEGRATION COEFFICIENTS				
		BRICK NO.	PERCENT	FIRST TREATMENT	SECOND TREATMENT	THIRD TREATMENT	FOURTH TREATMENT	FIFTH TREATMENT
Sand-Lime Bricks, Hardened in Steam	1912	1	11.82	B	F	—	—	—
		2	13.18	B	F	—	—	—
		3	12.61	B	F	—	—	—
		4	12.61	B	F	—	—	—
		5	13.14	B	F	—	—	—
		Mean	12.67					
Sand Cement Brick, Hardened by Setting	913	1	16.40	A	A	B	B	B
		2	20.97	A	A	B	B	B
		3	17.78	A	A	B	B	B
		4	18.50	A	A	B	B	B
		5	19.87	A	A	B	B	B
		Mean	18.70					

REMARKS. The very diverse behavior of these two samples is one of the most surprising facts brought out by the investigation. The photographs of sample 9 are shown in Figures 28-29.
The photographs of Sample 12 are shown in Figures 16-17.

Before entering upon a discussion of these results, it will be well to define the points at issue, upon which we must have answers before we can ask the public to accept and abide by the results of this sodium sulphate test. These are:

First. Has this treatment the power to disintegrate material of poor cohesive strength, while at the same time failing to disintegrate material of good cohesive strength?

Second. Assuming that when materials disintegrate in ordinary use, by the crystallization of water or solutions in their void spaces, the action is of the same general nature as that of the sodium sulphate freezing, is the latter so much more powerful or violent in its action as to make comparisons impossible or valueless?

Third. If the results of sodium sulphate freezing cannot be correlated with ice freezing, is the sodium sulphate process too violent in action to be of value as an independent mode of judging the resisting power of materials to disintegration?

Fourth. If not, can any correlation or equivalence be now established between stability under the sodium sulphate test and

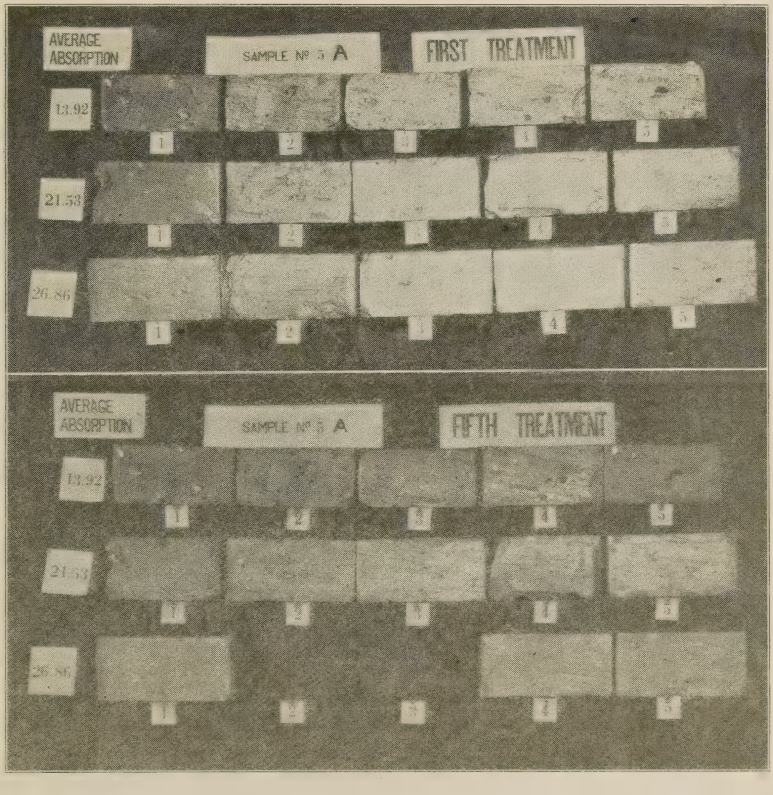


FIG. 24.

FIG. 25.

the other measurable properties of the material? If so, what are these relations?

Taking these up in order:

First. Has this treatment power to disintegrate materials of poor cohesive strength, while at the same time failing to disintegrate material of good cohesive strength?

The answer here is an unqualified yes. Tabulating the results in the order of their crushing strength, beginning with the highest and progressing to the lowest, and arranging the corresponding disintegration coefficients opposite, we have Table XLIII:

TABLE XLIII

MEAN CRUSHING STRENGTH ON EDGE	DISINTEGRATION COEFFICIENTS					
LBS. PER SQ. INCH	A	B	C	D	E	F
More than 15000....	5	..	..	..	..	..
15000-13000	..	..	..	..	..	..
13000-11000	5	..	..	..	..	..
11000-9000	25	..	..	..	..	..
9000-7000	9	..	1	..	..	..
7000-5000	15	..	..	..	..	..
5000-4000	19	2	4	..	..	..
4000-3000	3	..	4	1	7	..
3000-2500	8	..	7	..	..	..
2500-2000	..	1	1	..	..	3
2000-1500	3	..	..	3	11	13
1500-1000	2	..	..	..	2	6
Less than 1000.....	..	5	..	..	..	..
	94	8	17	4	20	22

An effort to plot this data does not add anything to our grasp of it, as there is not a large enough number of determinations to get smooth curves. But, it can be seen at a glance that the first actual rejections occur between 3000-4000 crushing strength (7 E) and the first actual failures as low as 2000-2500. Below 2500, the proportion of rejections and failures becomes suddenly large, showing that the process is selective in its operation.

Second. Is the violence of sodium sulphate freezing so much greater than ice freezing, that no parallels can be drawn? On this point, I have no new evidence to submit. A large number of ice freezing tests have been made in my laboratory, and under my direction in commercial plants, and the mode of failure of ceramic products by ice freezing is familiar to me. I see in the behavior of ceramic products in the two processes a very

close similarity of behavior, but a difference in degree of activity. I roughly estimate the sodium sulphate as about five times as active as ice. That is, twenty-five treatments with ice usually leave a varied group of materials in about the same state of demolition as five sulphate treatments.

But, until these two methods shall have been compared upon a large number of samples, using identical material, with all variables removed except that of mode of freezing, no one can make quantitative statements with precision or authority.

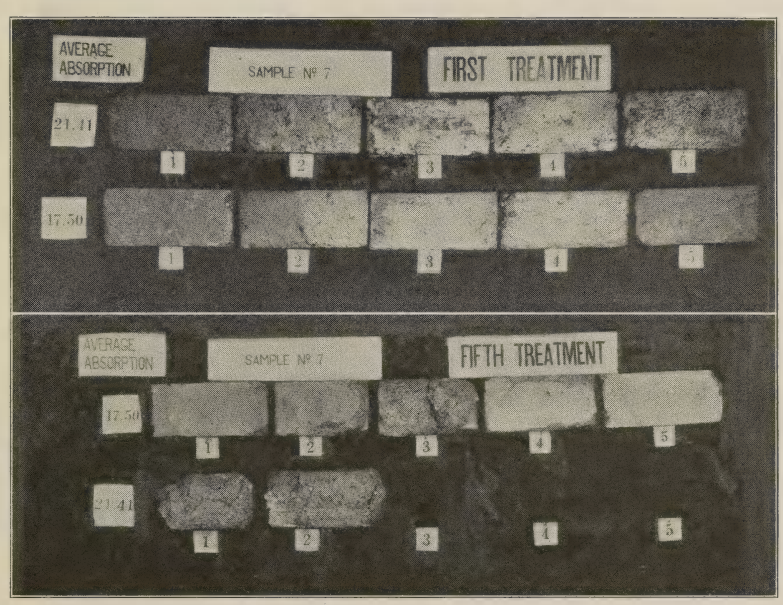


FIG. 27.

FIG. 26.

Third. Is the sodium sulphate process worth while in itself, regardless of its correlation with ice freezing?

In my opinion, it is. Even if prolonged study should fail to make exact correlation with the ice process, I submit that its greater severity than ice should be an advantage rather than a detriment. Any strength test is, in the nature of the case, more violent and sudden in its application than the strains which the material is expected to endure in natural use. The whole theory of strength testing is to explore the possibilities of a

material, to find out how much more a material will do than natural use requires, and thus to set suitable factors of safety for its use. A test, to be of any practical use, has to imitate the conditions of use as closely as possible, or at least use the properties which control in its actual use, but also has to concentrate in a relatively short period of time, a sufficient amount

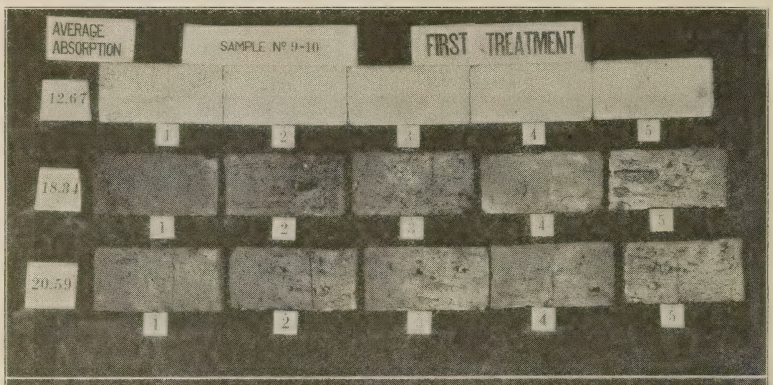


FIG. 28.

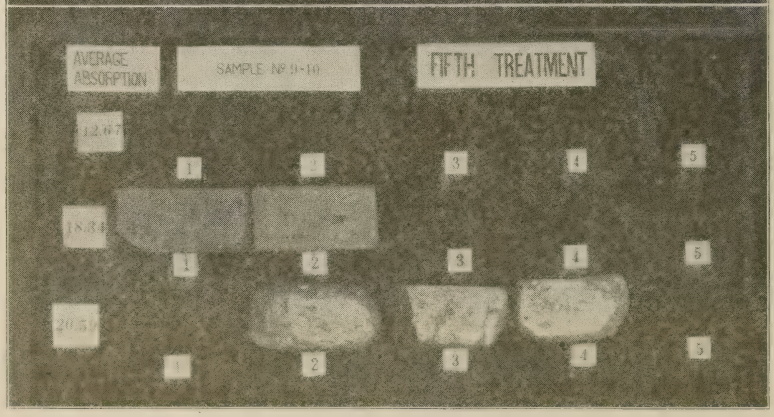


FIG. 29.

of wear, or strain or destructive condition to equal the normal life period of the product in question. A paving brick is expected to last twenty years or thirty years in the street, but a rattler test is expected to give in an hour, a decision as to whether the bricks will stand. A hoisting cable is expected to lift loads of ten tons in rapid succession for a year, before wear

reduces it too seriously for safety. But the testing machine must in a few minutes determine whether its initial ultimate strength is sufficient to stand these numberless repetitions of strain, with constant wear and corrosion reducing its effectiveness daily. Numerous illustrations of this sort might be given.

In the case of frost resistance, the question is not one of absolute immunity, but relative immunity. No building material is absolutely immune to attack of weather. All natural and artificial rocks succumb, in time, to the slow agencies of solu-

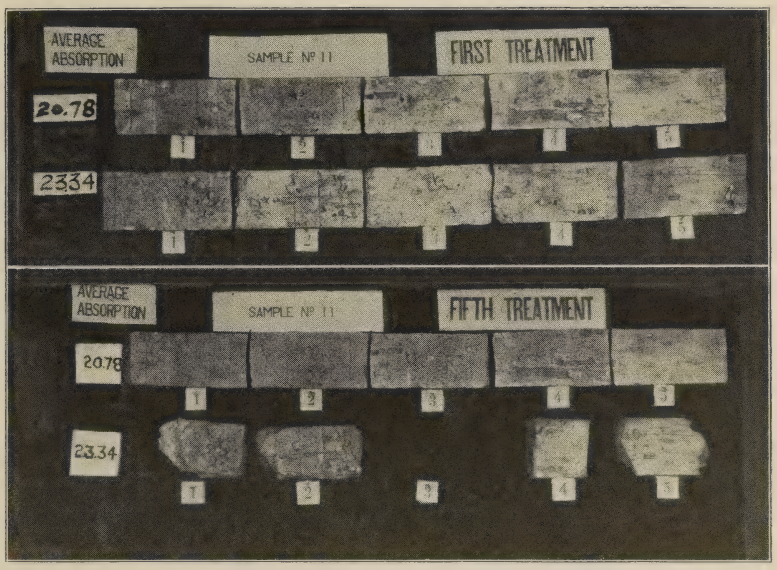


FIG. 30.

FIG. 31.

tion and frost. What we need to know is whether a given material has strength enough, under normal conditions of use, to resist frost and weather well, from the very finite human standpoint. Whether we depend upon twenty-five freezings with water, under a necessarily very artificial set of conditions, or upon five or some other number of freezings with sodium sulphate under wholly artificial conditions, we shall in any case have to judge whether the strength displayed is enough or not enough to warrant the use of the material beyond any reasonable doubt. In my opinion, it all comes down to a matter of judg-

ment in interpreting the data, no matter what method we use, and it is as easy to train our judgment to interpret the data of the sodium sulphate test as that of real freezing. Neither can be translated directly into terms of actual use. The sodium sulphate test has the great advantage of speed, simplicity, and cheapness, while ice freezing is slow, and in most cases of prohibitive cost.

Fourth. Can any correlation be now made between stability under sodium sulphate freezing and the other physical properties of bricks?

Two sets of facts only are available for comparison in the foregoing study, in crushing strength, and percentage of absorption.

Correlation of Crushing Strength and Sulphate Disintegration Coefficients. Some comparisons were given in another connection, on this head. Before trying to reach conclusions as to where to draw lines, even tentatively, we must take into consideration the fact that the process of manufacture has a vital influence in determining the resistance of a brick to frost, and it will be wrong to expect all bricks, regardless of what they are made of or how they are made, to conform to one standard. Referring back to Table XLIII and dividing it into two tables, stiff mud bricks and soft mud bricks respectively, we get Table XLIV.

TABLE XLIV

STIFF MUD BRICKS

MEAN CRUSHING STRENGTH ON EDGE	DISINTEGRATION COEFFICIENTS.					
LBS. PER SQ. INCH	A	B	C	D	E	F
Over 15000	5	..	..	..	..	..
15000-13000	..	..	..	..	..	..
13000-11000	5	..	..	..	..	..
11000-9000	25	..	..	..	..	..
9000-7000	9	..	1	..	..	..
7000-5000	15	..	..	..	..	..
5000-4500	6	2	2	..	..	..
4500-4000	3	..	2	..	..	..
4000-35000	..	..	..	..	5	..
3500-3000	..	..	..	..	..	..
3000-2500	3	..	7	..	..	..
2500-2000	..	..	..	..	..	..
2000-1500	..	..	..	3	4	3
1500-1000	..	..	..	..	..	..

SOFT MUD BRICKS

5000-4500	10	..	..	..	..	..
4500-4000	..	..	..	..	..	..
4000-3500	..	..	..	..	..	..
3500-3000	3	..	4	1	2	..
3000-2500	5	..	..	..	..	..
2500-2000	..	1	1	..	3	..
2000-1500	3	..	..	..	7	5
1500-1000	2	..	..	..	2	6

Correlating crushing strength with sodium sulphate freezing and considering these two factors only, the above data would enable us to say that 4,000 pounds would make a fairly safe strength requirement for stiff-mud bricks. As a matter of fact, out of 100 bricks, only five bricks having a crushing strength above 2,000 pounds would have to be rejected, while below 2,000 pounds, ten bricks would be rejected.

In the same way, for soft mud bricks, the actually necessary rejections, found from 2500 pounds down, number twenty-three, with only seven acceptances, while above 2500 pounds the rejections number three, and acceptances twenty-two.

In all probability 4,000 pounds for a stiff-mud requirement is no more severe than 2500 pounds for a soft-mud requirement.

Correlation of Absorption Data with Sodium Sulphate Freezing. Making a similar division into stiff-mud and soft-mud and tabulating the data, we get Table XLV.

TABLE XLV

STIFF MUD BRICKS

MEAN ABSORPTION PER CENT	DISINTEGRATION COEFFICIENTS					
	A	B	C	D	E	F
1.00-2.00	5	..	..	..	..	..
2.00-3.00	9	..	1	..	..	..
3.00-5.00	5	..	..	..	..	..
5.00-6.00	10	..	..	..	..	..
6.00-7.00	10	..	..	..	..	..
7.00-8.00	10	..	..	..	..	..
12.00-14.00	10	..	..	..	..	..
16.00-17.00	5	..	5	..	5	..
17.00-18.00	..	..	5	..	..	..
18.00-20.00	3	..	2	..	..	..
20.00-21.00	..	..	..	3	..	2
21.00-22.00	..	..	..	..	4	1

SOFT MUD BRICKS

13.00-14.00	8	2	..	..	..	..
17.00-18.00	..	..	2	1	2	..
18.00-19.00	..	1	1	..	..	3
19.00-20.00	5	..	..	..	..	..
20.00-21.00	5	..	..	..	3	2
21.00-23.00	3	..	2	..	2	3
23.00-24.00	2	..	..	..	4	4
26.00-27.00	3	..	..	..	..	2

From the above, we might say that the sodium sulphate test will attack and destroy or seriously injure a large majority of stiff mud bricks (especially if made of shale) whose absorption much exceeds 12 percent and that it will not seriously attack bricks of this sort whose absorption is below 12 percent.

We cannot make a similar generalized statement regarding soft-mud bricks. Inspection of Table XLV shows successes and failures promiscuously dotted over the whole chart. The re-

sisting power to internal expansion is evidently more a function of the clay itself, and less a function of the hardness of burning than in the case of shales. If we were to attempt to draw a line, it would be at 20 percent, though no clear case can be made so far as this data is concerned, for that or any other figure.

On the other hand, if we observe the power of the sulphate test to differentiate between soft-mud bricks of one make, burned to different degrees of hardness, some astonishingly sharp decisions are found.

For instance, in sample No. 3, group 2, we find with three treatments the sulphate attacks and destroys three bricks of 23.43, 24.50 and 26.26 percent absorption respectively, while two others of 20.28 percent and 22.35 percent endure five treatments without damage.

Again, in sample 5a, group 3, we find two bricks, 29.60 and 28.31 percent absorption respectively, failing promptly in the first and second test, while three others, 24.78, 25.21 and 26.41 percent, endure five tests with safety. The wonder is that any brick of such high porosity would stand the test at all.

Again, in sample 10, groups one and two, we find the dividing line between safety and failure apparently near 17 percent absorption. Of the 10 brick, of which the extreme range of absorption is a trifle more than 3 percent, the two lowest, 17.65 and 17.98 percent, have coefficients of B and C respectively in five treatments, while the 18, 19 and 20 percent material fails.

Again, in sample 11, groups 1 and 2, we have bricks ranging from 18.48 percent to 21.55 percent standing five treatments unscathed, while 22.79 percent and 22.94 percent begins to fail in three treatments, and 23.26 percent, 23.81 percent and 23.88 percent fail in two treatments.

The evidence is not all of this consistent character; we have some instances where a brick of high absorption stands better than another of lower absorption. In general, these differences are small, however, and may perhaps be explained by saying that between several bricks, all above the danger line, that one will fail first whose accidents of structure are most against it.

This absorption-disintegration study brings out what has been recorded before by various observers, viz., that it is impossible to correlate the other physical properties of clay products horizontally with their absorption, because this neglects the differences due to plasticity, fineness of grain, mineral composition, etc. While absorption is a powerful determining factor, it may well happen that a sandy clay, possessing in its finer grained bonding material, some very fusible mineral, may make a strong product with a high absorption, while another clay of equally sandy texture, and no low temperature fluxes, would be wholly untrustworthy at the same absorption percent, because not yet bonded by the flux in burning.

Correlation of the Sodium Sulphate Freezing Process with the Tentative Classification of Building Bricks Proposed by the A. S. T. M. The latter classification is founded upon a recognition of the above stated facts. It proposes that three different factors, absorption, crushing strength and modulus of rupture in cross breaking, shall be considered. If a brick of satisfactory absorption limit shows that its strength factors are bad, it is dropped below that grade in which it would be classified by absorption alone and into a grade which its strength entitles it to enter. Similarly, a brick of abundant physical strength might show a poor absorption test, and be actually dangerous in frosty exposures, and again the double standard would drop it in scale to a lower grade. The A. S. T. M. specifications (tentative) are given in Table XLVI.

TABLE XLVI — A. S. T. M. TENTATIVE SPECIFICATIONS

NAME OF GRADE	ABSORPTION LIMITS		CRUSHING STRENGTH (ON EDGE)		MODULUS LIMITS	
	MEAN OF 5	INDIV. MAX.	MEAN OF 5	INDIV. MIN.	MEAN OF 5	INDIV. MIN.
	percent	*percent*				
Vitrified Brick	5.0 or less	6.0	5000 or more	4000	1200 or more	800
Hard Brick	5.0 to 12.0	15.0	3500 or more	2500	600 or more	400
Common Brick	12.0 to 20.0	24.0	2000 or more	1500	450 or more	300
Soft Brick	20.0 and up	no limit	1000 or more	800	300 or more	200

SPECIFICATIONS PROPOSED TENTATIVELY FOR GRADING BY THE SODIUM SULPHATE PROCESS

Using the six symbols before given to express relative degrees of disintegration, and considering that the symbol C stands for only such attacks as have given no evidence of loss of internal strength, it is proposed to approve or pass any sample which shall show three or more A's with the two others not below C, and conversely to reject or fail any sample if any of its five bricks fall below C, or if three of them fail to attain the A grade. No intermediate rating is proposed.

Recasting the data heretofore given to meet the requirement of these two sets of specifications we have Table XLVII.

TABLE XLVII

Comparison between the Grading of 33 Samples of 5 Bricks Each
1 — By the A. S. T. M. Specifications
2 — By the Sodium Sulphate Freezing Process

SAMPLE DESIGNATIONS	ABSORPTION DATA		CRUSHING STRENGTH ON EDGE		DISINTEGRATION COEFFICIENTS						A. S. T. M. RATING	FREEZING TEST VERDICT
	MEAN FOR GROUP	INDIV. MAX.	MEAN FOR GROUP	INDIV. MIN.	A	B	C	D	E	F		
8-1	1.22	1.99	15139	11788	5	..	..	..	..	..		pass
2-1	2.05	2.52	7659	4646	4	..	1?	..	..	..	Vitrified	pass
6-1	2.74	3.94	10119	4530	5	..	..	..	..	..	Brick	pass
2-2	3.45	4.38	11283	7438	5	..	..	..	..	..		pass
1-1	5.23	6.14	8945	6250	5	..	..	..	..	..		pass
13-1	5.33	5.98	9029	6148	5	..	..	..	..	..		pass
6-2	6.06	7.61	9790	8965	5	..	..	..	..	..	Hard	pass
8-2	6.81	7.44	10878	9904	5	..	..	..	..	..	Brick	pass
13-2	7.24	7.36	9015	7527	5	..	..	..	..	..		pass
2-3	7.92	9.12	5934	4580	5	..	..	..	..	..		pass
4-1	12.23	13.75	5207	4191	5	..	..	..	..	..		pass
9	12.67	13.18	1912	1606	..	..	..	..	..	5		fail‡
8-3	12.74	13.55	5669	4737	5	..	..	..	..	..		pass
1-2	13.19	13.88	4693	3506	3	2	..	..	..	..		pass
5a-1	13.92	17.39	4876	4088	5	..	..	..	..	..		pass
6-3	16.09	17.35	3862	2795	..	..	..	..	5	..	Common	fail
5-1	16.12	17.15	4991	3142	3	..	2	..	..	..	Brick	pass
4-2	16.67	18.96	2835	1524	3	2	..	..	..	..		pass

7-1	17.50	18.09	3034	2748	..	..	2	1	2	..			fail
1-3	17.66	18.77	2580	1914	..	..	5	..	..	..			fail
5-2	18.40	20.00	4111	3120	3	..	2	..	..	..		pass	
3-1	19.27	19.96	2880	2376	5	..	..	..	..	..		pass	
10-2	20.59	20.88	1889	1378	..	..	..	..	3	2			fail
11-1	20.78	21.55	4465	3714	5	..	..	..	..	..		pass	
4-3	20.82	22.39	1508	844	..	..	..	3	..	2			fail
7-2	21.41	21.91	1421	1338	..	..	..	..	2	3	Soft		fail
5-3	21.43	21.77	1905	1571	..	..	..	..	4	1	Brick		fail
5a-2	21.53	23.41	3337	2632	3	..	2	..	..	..		pass	
11-2	23.34	23.88	1696	1171	..	..	..	..	4	1			fail
5a-3	26.86	29.60	1679	1365	3	..	..	..	..	2			fail
10-1	18.34	19.15	2168	777*	..	1	1	..	..	3	Unclassifiable		fail
12	18.70	20.97	913†	632	..	5	..	..	..	..	by		fail**
3-2	23.36	26.26	1126	399*	2	..	..	..	..	3	A. S. T. M.		fail

* Too low individual strength test.
† Too low mean strength test.
‡ Sand-Lime Brick.
** Cement Brick.

The preceding comparison will, it is thought, strengthen the argument for the more extensive study of the sulphate of soda freezing process. It does not seem to justify the fears of too great severity, entertained by Messrs. Howat and Bleininger, as its findings have agreed remarkably well with the other system of classification, which embodies the concensus of the best knowledge hitherto available on the classification of building brick.

It is significant that of the four lots of common bricks, which constitute the first four failures by the sulphate test, one is a sand-lime brick, which failed absolutely on the second freeze; two are very soft-burned stiff-mud shale bricks, which have not yet developed their bond, and the fourth is a soft-mud brick of conspicuously bad structure, being full of stones and coarse lime pebbles. In the opinion of the writer, these bricks all failed because they deserved to fail, and they belong in use, if used at all, in the next lower category, soft bricks, for use in interior walls and backing up purposes.

Similarly, the two lots of bricks graded as soft by the A. S. T. M. specifications, but which pass the sodium sulphate freezing test, are both soft-mud bricks which develop a good strength while still very porous, as discussed earlier.

By the A. S. T. M. specifications alone, such bricks would be condemned to inferior uses only, on account of their absorption percent. But, the freezing test shows that the high absorption in this case is no menace, and the bricks ought to go up into the common brick grade, being as good in every way as many that are so graded.

Another interesting point may be gleaned from considering lots 5-3 and 5a-2. These bricks are made from the same clay, on the same yard, No. 5 being stiff-mud and 5 A soft-mud. Their mean absorptions are the same, 21.43 and 21.53 respectively. But their crushing strengths are very different, 1905 and 3337 respectively. This is due to the lamination of the stiff-mud bricks, which ruined their crushing strength, and caused them to peel open in layers in the sulphate freezing, while the soft-mud structure, of exactly the same absorption percentage, enabled the sample to pass.

In conclusion, the writer desires to express the opinion first that the method of testing brought to the attention of this Society last year by the Bureau of Standards is an important addition to our means of studying the structure of ceramic products, and second that its severity is not too great to disqualify it, and third that its findings thus far seem to afford a wise decision or explanation for those cases in which the other specifications are in disagreement, and fourthly, the test should be applied to a much larger quantity and variety of material, with a view to using it in the specifications for testing and classifying building brick.

THE RELATIVE ACTION OF ACIDS ON ENAMEL—II

BY EMERSON P. POSTE

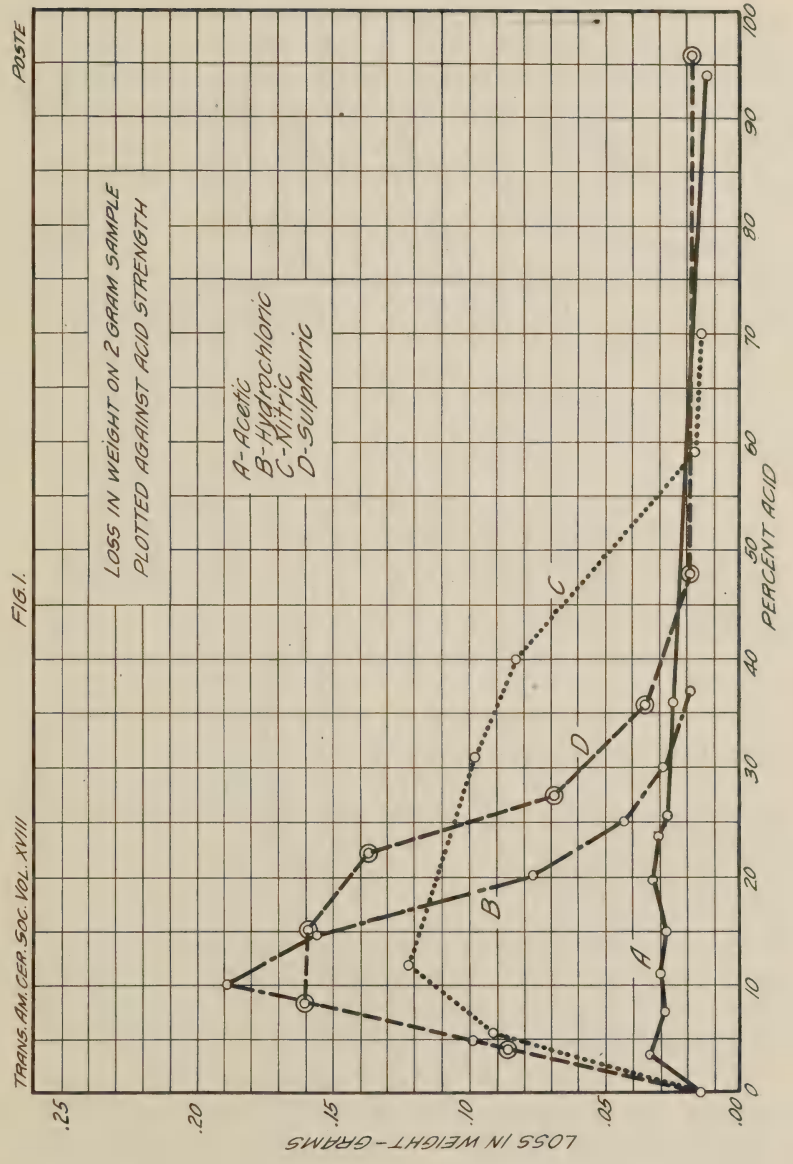
The paper under the above title in Volume XVII of the *Transactions* of this Society presented a method for determining the relative action of acids on an enamel, together with certain preliminary data obtained by that method.

The method used was that of determining the loss in weight of a 2 gram sample of pulverized frit due to the action of the solution under consideration. The residue, after exposure under standard conditions—24 hours in 100 cc. of solution at constant temperature of about 70° F.— was caught on a weighted Gooch crucible, and the loss of weight determined. The frit used was obtained by dry grinding in a small pebble mill and rejecting that portion retained on a 20 mesh screen. The data presented is summarized in Figure 1.

The general conclusions drawn were that the method had merits as a means of comparing the relative action of various strengths of a given solution on a given enamel, and of comparing the action of different compounds on a given enamel. It was also suggested that the action of a given substance in varying strengths of solution was in a general way proportional to the ionic concentration of the solution.

The discussion of the paper brought out the opinion that the method had no value as a means of comparing the relative resistance of different enamels. It was suggested that it would be better to narrow the size of particles to those passing one size mesh and retained on a finer mesh. The idea of the relation of the ion concentration to the chemical action was corroborated by curves showing the relation of ion concentration and conductivity.

The scope of the present paper is two-fold, to report the result of work suggested by the discussion of the former paper, and to show the effect of temperature on the solubility of enamel.


TRANS. AM. CER. SOC. VOL. XVIII
FIG. 1.
POSTE
LOSS IN WEIGHT ON 2 GRAM SAMPLE
PLOTTED AGAINST ACID STRENGTH
A-Acetic
B-Hydrochloric
C-Nitric
D-Sulphuric
LOSS IN WEIGHT - GRAMS
.25
.20
.15
.10
.05
.00
0
10
20
30
40
50
60
70
80
90
100
PERCENT ACID
A
B
C
D

In following out the suggestion of the use of particles within a narrow size limit, it became necessary to determine whether uniform results could be obtained under different conditions of fineness of the frit as a whole. In other words, would particles passing an 80 mesh and retained on a 100 mesh give the same results when taken from a sample ground for a certain time as when taken from a sample ground twice as long?

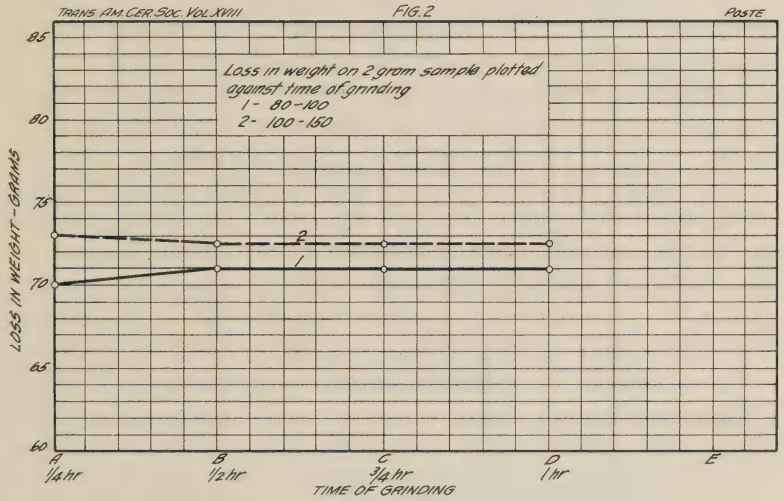
To determine this point four 1200 gram samples of frit of a comparatively low resisting enamel were ground in a pebble mill for ¼, ½, ¾ and 1 hour. These are designated as A, B, C, and D respectively. Fineness determinations gave the results shown in Table I.

TABLE I

MESH OF SIEVE	A	B	C	D
Retained on 20.....	0.3	0.1	0.0	0.3
Passed 20 — retained on 40.	20.5	8.4	2.5	0.8
Passed 40 — retained on 60.	22.1	14.1	4.1	0.9
Passed 60 — retained on 80.	6.6	3.1	3.1	0.9
Passed 80 — retained on 100.	9.0	10.5	6.2	3.7
Passed 100 — retained on 150.	13.4	16.6	16.0	13.5
Passed 150 — retained on 200.	6.8	11.1	13.0	12.3
Passed 200	21.0	36.1	55.1	67.6

For use, the portions passing 80 mesh and retained on 100 mesh were selected for one series of tests and those passing 100 and retained on 150 for another. Two grams of each were placed in 100 cc. of 11.8 percent hydrochloric acid and allowed to stand 24 hours at 70°F. The losses were as shown in Figure 2. The solid line designates the 80-100 mesh samples and the broken line the 100-150 mesh. It is seen that the losses are reasonably constant for various periods of grinding though the percentages for the 80-100 were 9.0, 10.5, 6.2 and 3.7, and for the 100-150, 13.4, 16.6, 16.0 and 13.5. Other data which have been obtained are consistent with the above.

Taken as a whole, this series of tests leads to the conclusion that the use of a narrow limit of particle size would not introduce variations due to differences of average fineness of the entire sample which would materially effect the results.


TRANS. AM. CER. SOC. VOL XVIII
FIG. 2
POSTE
Loss in weight on 2 gram sample plotted against time of grinding
1 - 80-100
2 - 100-150
LOSS IN WEIGHT - GRAMS
85
80
75
70
65
60
2
1
A
1/4 hr
B
1/2 hr
C
3/4 hr
D
1 hr
E
TIME OF GRINDING

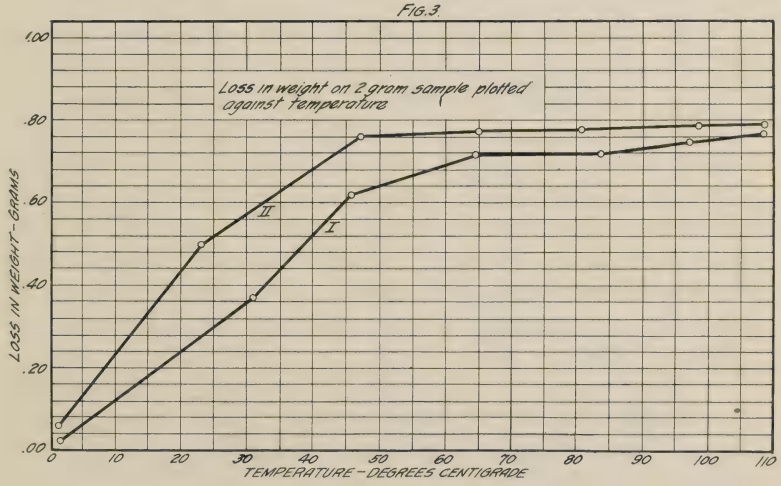


FIG. 3.
Loss in weight on 2 gram sample plotted against temperature
LOSS IN WEIGHT - GRAMS
1.00
.80
.60
.40
.20
.00
II
I
0
10
20
30
40
50
60
70
80
90
100
110
TEMPERATURE - DEGREES CENTIGRADE

To investigate the effect of temperature on the action of an acid on an enamel the following tests were made. Having used hydrochloric acid in the previous work, it seemed logical to continue its use. It was necessary first to determine a means of keeping it at constant strength under different temperatures. A series of trials showed that solutions under 20 percent in strength can be boiled under a reflux condenser without appreciable change of concentration during a period of four hours.

The first sample used was that obtained by mixing the 100-150 mesh portions of A, B, C, and D above. It was designated as I. The second sample, II, was obtained by grinding a sample of frit from another formula and separating the 100-150 mesh portions.

Each result was obtained by placing two gram samples in a flask containing 13.6 percent hydrochloric acid under a reflux condenser, the acid having been cooled or heated to the desired temperature. The enamel was recovered on a Gooch crucible after four hours exposure at constant temperature. Figure 3 shows the curves obtained by plotting the loss in weight against the temperature.

The results lead to the conclusion that by using a narrow limit of size of particles, reasonably uniform results can be obtained under varying conditions of fineness. This suggests a possible standard method of comparing the acid resistance of different enamels which would not necessitate grinding samples to the same general fineness. To keep a uniform area of surface it would be necessary to correct the sample weight for varying specific gravities. The effect of differences of mill additions and burning might seriously interfere with the results if used to judge enamels manipulated under different conditions. These points are yet to be worked out.

Finally, it is of interest to note that the action of an acid on an enamel varies quite markedly with the temperature, the gradient decreasing quite rapidly as the temperature approaches the boiling point of the acid.

KAOLIN REFINING

BY IRA E. SPROAT

Introduction. The question—"Why can not American kaolins be substituted for English china clay?"—has been asked time and time again, and more often since the beginning of the present European war which has threatened to cut off the supply of European clays. The answer to this question has invariably been, that the domestic kaolins are not as pure and uniform in composition as the imported clays.

This demand for white-burning American clays is of such economic importance that it was considered advisable to determine whether some of our vast deposits of impure white clays could not be refined sufficiently to permit their being substituted for the foreign materials.

Up to the present time the application of only mechanical principles have been made use of in the improvement of methods of refining kaolins, but in order to keep pace with the ever increasing requirements for better quality and uniformity of product, the application of the principles of colloidal chemistry is necessary. The work represented by this report was carried on for the purposes of determining the practicability of the application of the technical control of clay disperse systems in the refining of kaolins and the utilization of the treated clay in the manufacture of vitreous china and wall tile.

It is believed that this report will prove stimulating to the clay washing industry, will point the way to improvements in methods of refining, and will lead to a large use of domestic kaolins by manufacturers of high-grade ceramic wares.

Kaolin Refining in the Dry Branch District. Only one company (The Georgia Kaolin Co.) in this district is actively engaged in the refining of kaolin. The method of washing clay at this plant is very similar to the refining process used in the refining of the primary kaolin of the Southern Appalachian district,

which has been described in detail by Prof. Arthur S. Watts (Page 72, *Bulletin No. 53, U. S. Bureau of Mines*), so only a mere outline of the washing process will be given here. It consists of six different operations which are as follows:

1st. A thorough blunging of the clay with water. This is done in rectangular blungers which are about 10 feet long, 4 feet wide and 4 feet deep.

2d. The floating of the slip through long narrow troughs. These troughs are about 50 feet long with an average width of about two feet, the width of trough depending upon the rate of flow required to settle out the impurities.

3d. Screening of the slip through a 110-mesh rotary screen.

4th. Concentrating the slip in large concrete tanks which are about 75 long, 25 feet wide and 5 feet deep, where it is allowed to stand for about 12 hours, when the supernatant liquid is drawn off, and the thickened clay slip run into a small retaining tank.

5th. Filter pressing the thick clay slip which is pumped from the small retaining tank into the filter at a pressure of 100 pounds.

6th. Drying the filter press cakes which contain about 20 percent of water in tunnel dryers heated by exhaust steam.

7th. Crushing the thoroughly dried press cakes into small pieces in corrugated rolls.

Impurities in Georgia Kaolins. The following is a chemical analysis of a sample of kaolin taken from the pit of the Georgia Kaolin Company and reported by S. W. McCallie (Page 128, *Bull. 18, Ga. Geol. Surv.*):

	percent
Moisture at 100°C	1.22
Loss on ignition	13.46
SiO_2	44.76
Al_2O_3	38.41
Fe_2O_3	0.63
CaO	0.20
MgO	0.09
Na_2O	0.09
K_2O	0.35
TiO_2	1.37

From this analysis it is seen that the chief color-imparting oxides are Fe_2O_3 and TiO_2. The source of these oxides is best shown by the following micrscopic examination of a sample of clay from the mine of the George Kaolin Company.

Examinations made by F. B. Laney of the Bureau of Mines.

Quartz.
*Partially decomposed feldspar often stained with iron oxide.
*Wad—small pisolites of manganese oxide.
*Limonite.
Muscovite.
*Magnetite.
*Hematite.
*Ilmenite.
Zircon.
Rutile.
Apatite.
*Tourmaline.
Corundum.
Monazite.
*Iron—minute particles broken off from the crushing machinery.

The minerals marked * are the minerals which tend to give the cream color to a burned china body when Georgia kaolin is one of the constituetns of the body.

Only a portion of these troublesome minerals are removed by the present methods of refining, due to the fact that the most of the color imparting minerals are present in a very fine state of subdivision, as the coarser impurities settled out when the clay was being deposited. In fact most of the color imparting impurities present will pass a 260-mesh sieve, and some are much finer.

Besides being finely subdivided, these impurities are completely coated with clay which prevents them from being settled out (regardless of their higher specific gravity) without a big loss of clay. This clay coating is not removed to any great extent on prolonged blunging. Even blunging for two hours will not dis-

perse the clay particles. However, grinding in a ball mill has a greater tendency to produce complete dispersion, but this sort of treatment is not an economical one to use in the washing of kaolins. Therefore, in order to obtain complete dispersion of the clay grains and the resulting separation of the color imparting minerals from the clay it is necessary to utilize the principles of colloidal chemistry.

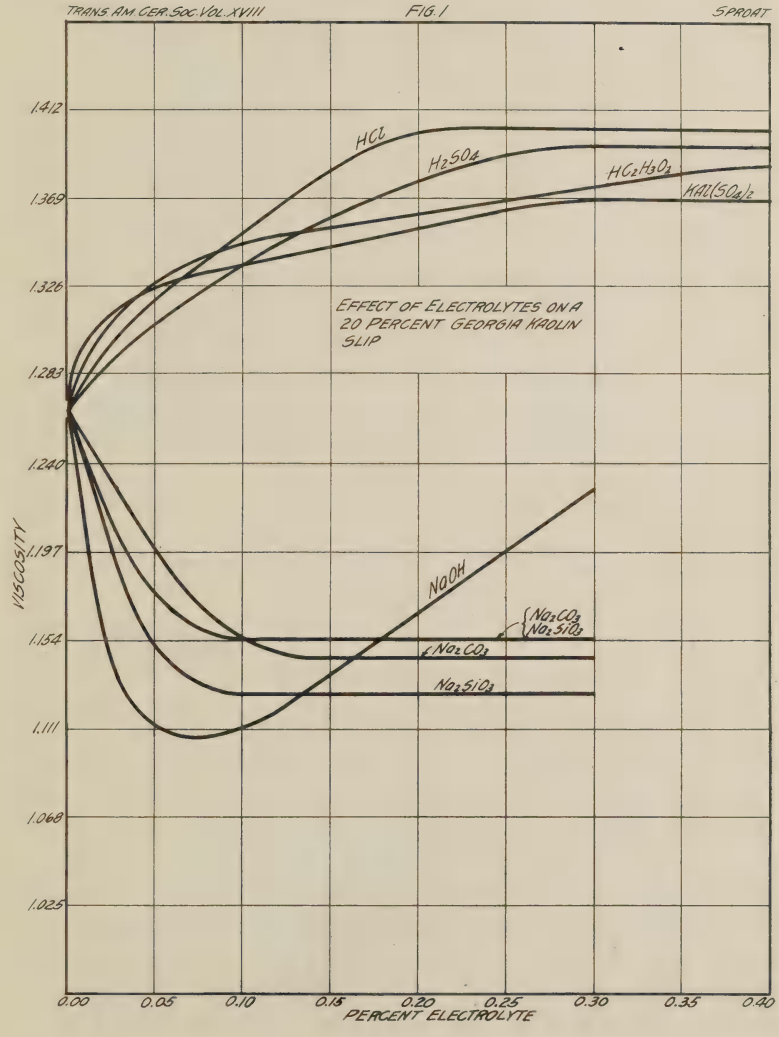
EFFECT OF ALKALIS, ACIDS AND SALTS UPON GEORGIA KAOLIN WATER SYSTEMS

Much work has been done in this country and abroad on the effect of electrolytes on clay suspensions, and in nearly every investigation carried on in this country, Georgia kaolin was included among the clays tested. Therefore, only a brief summary of the effect of alkalis, acids and salts upon Georgia kaolin suspensions will be given here:

Effect on Viscosity. It is quite well understood by this time that alkalis in small amounts tend to increase the state of dispersion of clay-water systems due to the electrostatic repulsion of the negatively changed O H ions, the result being a decrease in viscosity of the system. This breaking up of the larger clay particles into smaller ones is plainly shown by the following mechanical analyses of two samples of Georgia kaolin, one of which contained 0.075 per cent NaOH. These analyses were made on a Schultze elutriation apparatus with an overflow of 80 c.c. per minute. (See page 46, *Bull 53, U. S. Bureau of Mines*).

TABLE I

SEPARATES	WASHED GEORGIA KAOLIN	WASHED GEORGIA KAOLIN CONTAINING 0.075 PERCENT NaOH
200 mesh	0.27	0.28
260 mesh	0.42	0.45
330 mesh	0.33	0.30
No. 1 jar	4.40	0.92
No. 2 jar	2.36	2.30
No. 3 jar	15.70	9.04
No. 4 jar	76.52	86.61


TRANS. AM. CER. SOC. VOL. XVIII
FIG. 1
SPROAT
1.412
1.369
1.326
1.283
1.240
1.197
1.154
1.111
1.068
1.025
VISCOSITY
HCl
H2SO4
HC2H3O2
KAl(SO4)2
EFFECT OF ELECTROLYTES ON A 20 PERCENT GEORGIA KAOLIN SLIP
NaOH
Na2CO3
Na2SiO3
0.00
0.05
0.10
0.15
0.20
0.25
0.30
0.35
0.40
PERCENT ELECTROLYTE

From this table it is seen that the particles which remain upon a 330-mesh sieve are little affected by NaOH, therefore they are chiefly impurities and not colloidal clay.

On the other hand, acids and salts operate to produce the oposite effect from that of alkalis, causing a coagulation of the clay particles and a resulting increase in viscosity of the system.

Therefore, the effect of the addition of different amounts of electrolytes to a clay-water system can be satisfactorily studied by the determination of viscosity. This is best done by use of a brass efflux tube or viscosimeter. The viscosimeter used by the writer is very similar to the one described by Prof. A. V. Bleininger (Page 20, *Technologic Paper No. 51, U. S. Bureau of Standards*) with the exception of the size of the efflux which is 3/32 instead of 5/32, as recommended by Prof. Bleininger. This reduction in size of efflux is necessary when determining the viscosity of slips containing a small percentage (less than 25 percent) of solids. All viscosity determinations made by the writer are based on the volume of water that ran through the 3/32 efflux tube in 117 sec. which is to be understood when mention is made of viscosity determinations throughout this report. The viscosity of all clay suspensions are computed in terms of water by dividing their observed time of flow by 117.

Figure 1 shows the effect of alkalis, acids and salts upon the viscosity of a Georgia kaolin slip containing 20 percent clay. From these curves it is seen that NaOH shows the greatest drop in viscosity reaching a well defined minimum at 0.075 percent addition of the reagent. Upon further additions of caustic soda, the viscosity at once increases. In the case of Na_2SiO_3 and Na_2CO_3 the decrease in viscocity is not as great as that caused by additions of NaOH. Besides the minimum viscosity is not reached until 0.10 percent of sodium silicate and 0.125 percent of sodium carbonate are added to the clay water system. A mixture of 50 percent Na_2CO_3 and 50 percent Na_2SiO_3 gives a less drop in viscosity than either of the reagents, the minimum viscosity being reached with 0.10 percent addition of the mixture. Neither sodium carbonate, sodium silicate or a mixture of the two show a tendency towards increasing the viscosity.

In the case of the coagulating agents HCl shows the greatest tendency along this line, reaching a well defined maximum viscosity upon the addition of 0.2 percent of the reagent, while acetic acid and alum do not increase the viscosity as much as hydrochloric acid. The maximum viscosity produced by acetic acid and alum is 1.384 and 1.376 respectively, as compared to 1.401 that produced by HCl, but these reagents are much more active in coagulating the clay particles below 0.05 percent additions. Besides this, the addition of 0.4 percent of acetic acid and 0.3 percent alum is necessary to reach the maximum viscosity. In the case of H_2SO_4 the maximum viscosity of 1.393 is reached with the addition of 0.275 percent of the reagent.

Effect upon Sedimentation. It is also a well known fact that additions of small amounts of alkalis tend to hold the clay in suspension or in a state of deflocculation which corresponds to decreasing viscosity, while acids and salts have the opposite effect upon the clay-water system, causing a more rapid settling of the clay particles. The clay-water system is then said to be in a state of flocculation, which corresponds to an increase of viscosity.

THE FUNCTIONS OF ELECTROLYTES IN THE REFINING OF KAOLINS

History. Ashley[1] found that the color of clays in which iron is one of the bases would be improved if the clay were blunged with HCl, settled, and the overlying water drawn off.

Ashley[2] also reports Keppler's patent on improving clays by, first, deflocculating with alkali, second, making an addition of colloid matter from humus, third, reprecipitating all together by the addition of an acid.

Bleininger[3] in summarizing his results on the electrical separation of clay, says the use of caustic soda and other electrolytes brings about a condition of minimum viscosity which greatly assists in the separation of quartz and feldspar.

Brown and Howat[4] found that by defiocculating kaolins with caustic soda or silicate of soda their color was improved

[1] *Trans. Amer. Cer. Soc.*, Vol. XII (1910), p. 802.
[2] *Trans. Amer. Cer. Soc.*, Vol. XII (1910), p. 803.
[3] *Trans. Amer. Cer. Soc.*, Vol. XV (1913), p. 343.
[4] *Trans. Amer. Cer. Soc.*, Vol. XVII (1915), p. 87.

and the drying shrinkage decreased, but the burning shrinkage increased.

Prof. Bleininger[5] states: "that the washing of kaolinitic clays may be decidedly improved as far as the quality of the product is concerned by adding to the water in the blunger a definite small amount of caustic soda or a mixture of caustic soda and sodium silicate." This treatment improves the color of most clays, enriches their content in clay substance, decreases the drying shrinkage, but tends to increase the burning shrinkage.

General Principles. The fact that alkalis when added to clay-water systems increase the dispersion of the system with the resulting decrease in viscosity and increased suspension of the clay particles makes this principle of colloidal chemistry directly applicable to the purification of Georgia kaolins. The impurities in the Georgia kaolins are coated with clay which fact makes it impossible to settle them out without a great loss of clay, but when the clay-water system is dispersed these impurities are then free to settle out. While the consequent increased suspension of the clay particles gives a longer time in which the impurities have to be settled out there before the process is not economical, due to the settling of the clay. The success of any kaolin refining method making use of these principles of colloidal chemistry depends upon the technical control of the viscosity of the clay-water system. Therefore a study of the factors affecting both the initial and the minimum viscosity was made under the conditions of a present day washing plant.

FACTORS AFFECTING THE INITIAL VISCOSITY

It is a well known fact that present day kaolin refining methods are not only wasteful but very inefficient, which is shown very clearly by viscosity tests. These tests do not only point out the cause of inefficiency, but show why better results have not been obtained.

Varying Clay Content. Without some mechanical means of controlling the amount of clay going into the washer,

[5] *Tech. Paper Bureau of Standards,* No. 51, page 38.

TABLE II

DATE	HOUR	PERCENT WATER	PERCENT CLAY
March 20	9–0	90.0	10.0
24	10–0	91.6	8.4
	11–0	92.7	7.3
25	9–0	93.6	6.4
	10–0	90.2	9.8
	1–0	93.6	6.4
26	8–0	92.4	7.6
	10–0	93.6	6.4
	12–0	94.0	6.0
27	8–0	92.0	8.0
	10–0	91.8	8.2
29	8–0	94.8	5.2
31	11–30	95.6	4.4
April 1	8–00	91.7	8.3
2	9–30	93.0	7.0
3	2–45	95.5	4.5
5	7–30	99.4	0.6
	8–00	92.2	7.8
	9–00	94.2	5.8
	10–0	88.7	11.3
	1–30	90.9	9.1
	1–45	94.7	5.3
	5–00	91.8	8.2
7	7–15	91.8	8.2
	8–15	95.4	4.6
	9–45	93.9	6.1
	10–00	96.0	4.0
	11–00	94.4	5.6
8	6–45	91.8	8.2
	8–15	88.9	11.1
	8–30	94.2	5.8
	8–45	94.5	5.5
	1–30	91.5	8.5
9	8–30	88.9	11.1
	10–00	92.7	7.3
	2–00	91.8	8.2
10	8–00	90.5	9.5
	9–00	87.3	12.7
	10–30	93.9	6.1
	11–30	89.4	10.6
12	7–15	86.5	13.5
14	6–30	93.4	6.6
	8–00	91.4	8.6
15	5–00	88.6	11.4
16	10–00	89.6	10.4
	10–30	95.2	4.8
	1–00	91.8	8.2
	4–00	91.2	8.8

Average — 7.64 percent clay.

wide variations in percentage of clay in the slip will occur. These variations are sometimes very great, as is shown in Table II. From the data in this table it is seen that the percentage of clay varied from 0.6 percent to 12.7 percent, with an average of 7.64 percent for the month. These tests were made at all hours of the day and under varying conditions of weather, temperature, mining operations, and other changing factors, so that the average of 7.64 percent of clay represents the average conditions found in the present day washing plant. It can be readily seen that this wide range in composition of slip will affect both the quantity and quality of the product.

Degree of Blunging. The effect on the initial viscosity of different degrees of blunging is shown in curves Nos. 2, 3 and 4 of Fig. 2. Curves 3 and 4 are plotted from viscosity tests upon slips taken direct from the washer, the test of Curve No. 3 being made in dry weather and those of No. 4 in wet weather. From these two curves it is seen that there is a big increase in viscosity during wet weather. This increase is due to the fact that the clay is wet and sticky as it enters the washer which in turn decreases the blunging action of the washer, causing a decrease in the dispersion of the clay particles with the consequent increase in viscosity. It is a well known fact around kaolin refining plants that the clay will not settle as quickly in wet weather as in dry weather, and alum is used to precipitate the clay. This fact seems to be contradictory to the above statements, but the reason for the slow rate of settle is not due to any increased dispersion, but to the decreased percentage of clay in the slip.

The effect of increased blunging action is shown in Curve No. 2 of Fig. 2, the slips for this test being made up from clay taken direct from the mine during dry weather, blunged in a ball mill for two hours, screened through a 120-mesh sieve and then tested. From this curve it is seen that the increased blunging causes an increased drop in the viscosity due to increased dispersion.

Predrying. Curve No. 1, Fig. 2 shows the effect of drying clay before blunging it up with water. These tests were made by blunging the bone-dry clay with water in a small house-

TRANS. AM. CER. SOC. VOL. XVIII
FIG. 2.
SPROAT
EFFECT OF ALUM, DEGREE OF SLUNGING AND PRE-DRYING ON GA. KAOLIN SLIPS
VISCOSITY
PERCENT CLAY
1.455
1.412
1.369
1.326
1.282
1.240
1.197
1.154
1.111
1.068
1.025
0.982
1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18 19 20
1
2
3
4
5
Clay predried - before slunging
After 2 hours slunging
Clay from washer
Clay from washer in wet weather
Water containing 0.3 percent alum

hold churn. It is seen from this curve that drying produces a still greater degree of dispersion with the resulting drop in viscosity.

Alum. The tests represented by Curve No. 5, Fig. 2, were made in dry weather, but the feed water contained about 0.3 percent of alum. From this curve it is evident that small percentages of alum will greatly increase the viscosity of the clay-water system. The effect of the alum becoming more pronounced as the percentage of solids in the slip increases.

THE EFFECT OF VARYING VISCOSITY ON CAPACITY OF A REFINING PLANT

The capacity of a washing plant depends upon the maximum flow of water through the washer and the percentage of clay held in suspension at the maximum viscosity permissable without retarding the precipitation of the impurities. This maximum permissable viscosity will vary to some extent with the different clay deposits and can only be determined by close observations of a number of settling tests of slips of varying viscosities. In most cases a viscosity of about 1.094 will be the maximum. However, it is advisable to run at a viscosity a little lower than the maximum, a viscosity of 1.068 being very safe practice.

The maximum amount of water that will flow through the ordinary rectangular washer, 10 ft. by 4 ft. by 3 ft, without overflowing, and produce the maximum degree of dispersion under given blunging conditions depends upon the condition of the clay entering the washer, whether wet or dry, lumpy or disintegrated. Under the present day conditions of kaolin refining, where the clay goes into the washer in large lumps, the maximum flow is about 1200 cu. ft. per hour.

It has been shown that a number of factors influence the percentage of clay contained in a slip of a given viscosity. Therefore these factors tend to increase or decrease the capacity of a washing plant, according to whether they decrease or increase the viscosity of the clay-water system. For example, suppose the plant was operating under dry weather conditions and maintaining a constant viscosity of 1.094, the slip would then con-

tain 10.8 percent of clay, but under wet weather conditions the slip would only contain 7.4 percent of clay, a decrease in capacity of 31 percent, while on the other hand if the clay had first been dried the slip would contain 16.2 percent of clay, an increase in capacity of 50 percent over dry weather conditions.

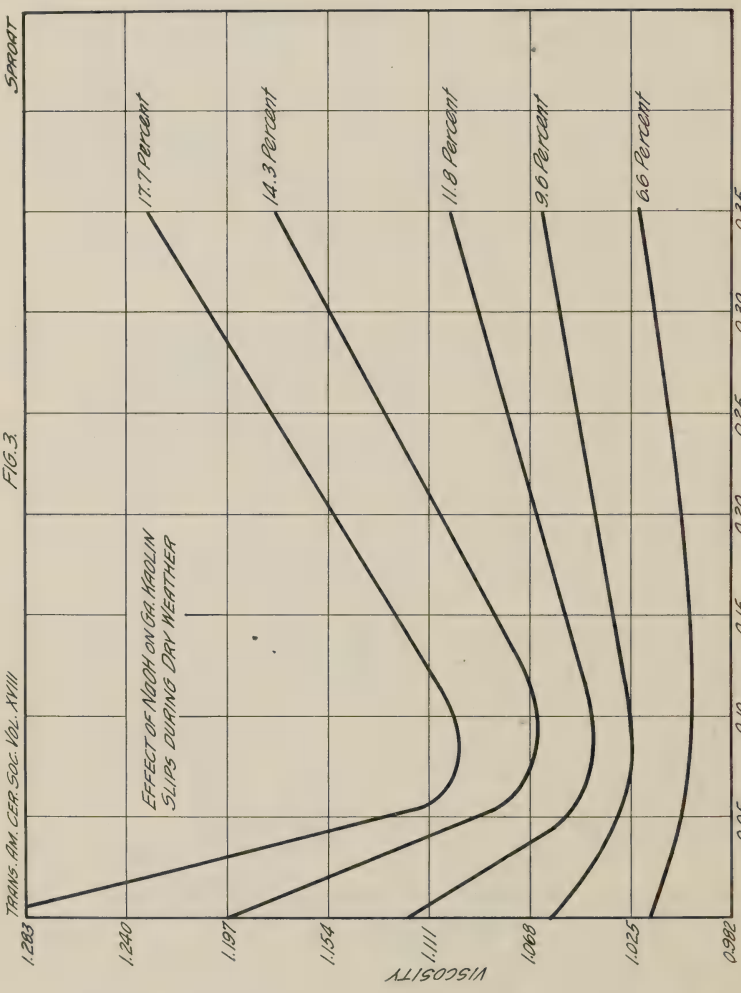
FACTORS AFFECTING THE MINIMUM VISCOSITY

Since caustic soda produces the greatest dispersion of Georgia kaolin-water systems, it was used in the commercial tests on kaolin refining, and it was found that only some of the factors affecting the initial viscosity had any effect or influence upon the degree of dispersion or the minimum viscosity produced by small additions of alkalis.

Degree of Blunging. It is seen upon comparing the diagrams of Fig. 3, made in dry weather with those of Fig. 4, made in wet weather, that the degree of dispersion or minimum viscosity of the slips of the same clay content is the same regardless of the wide difference in initial viscosity. For example: From the curve in Fig. 3, representing a slip of 9.6 percent clay it is seen that the initial viscosity is 1.059 and the minimum 1.005, while in Fig. 4, a slip of 9.4 percent solids shows an initial viscosity of 1.145, but the same minimum of 1.025.

In comparing the curves of Fig. 5 which were plotted from tests of slips previously ground in a ball-mill with those of Fig. 3, similar conditions prevail. Although there is a big drop in initial viscosity, the minimum viscosity is the same for slips containing the same percentage of clay. Therefore the extent of blunging has no effect upon the degree of dispersion or minimum viscosity of a slip of given clay content.

Predrying. The curves in Fig. 6 are plotted from tests upon slips of Georgia kaolin which had been previously dried to bone dryness on the top of steam pipes. By comparing the curves of Fig. 6 with those of Fig. 3, it is seen that predrying of the clay decreases the initial viscosity (in slips containing less than 15 percent clay) to that of the minimum viscosity. In other words, when the slip contains 15 percent or less of clay predrying is just as effective in increasing dispersion as is NaOH, but predrying is far more expensive than the use of


SPROAT
FIG. 3
TRANS. AM. CER. SOC. VOL. XVIII
EFFECT OF NaOH ON GA. KAOLIN
SLIPS DURING DRY WEATHER
17.7 Percent
14.3 Percent
11.8 Percent
9.0 Percent
6.6 Percent
VISCOSITY
1.283
1.240
1.197
1.154
1.111
1.068
1.025
0.982
0.05
0.10
0.15
0.20
0.25
0.30
0.35
PERCENT NaOH

NaOH. However, upon adding caustic soda to such slips the viscosity is still decreased further, but the drop being very small.

Since the minimum viscosity is not changed by the degree of blunging, and only slightly by very severe predrying conditions, the capacity of a plant using a dispersing agent will be constant under such changing operating conditions.

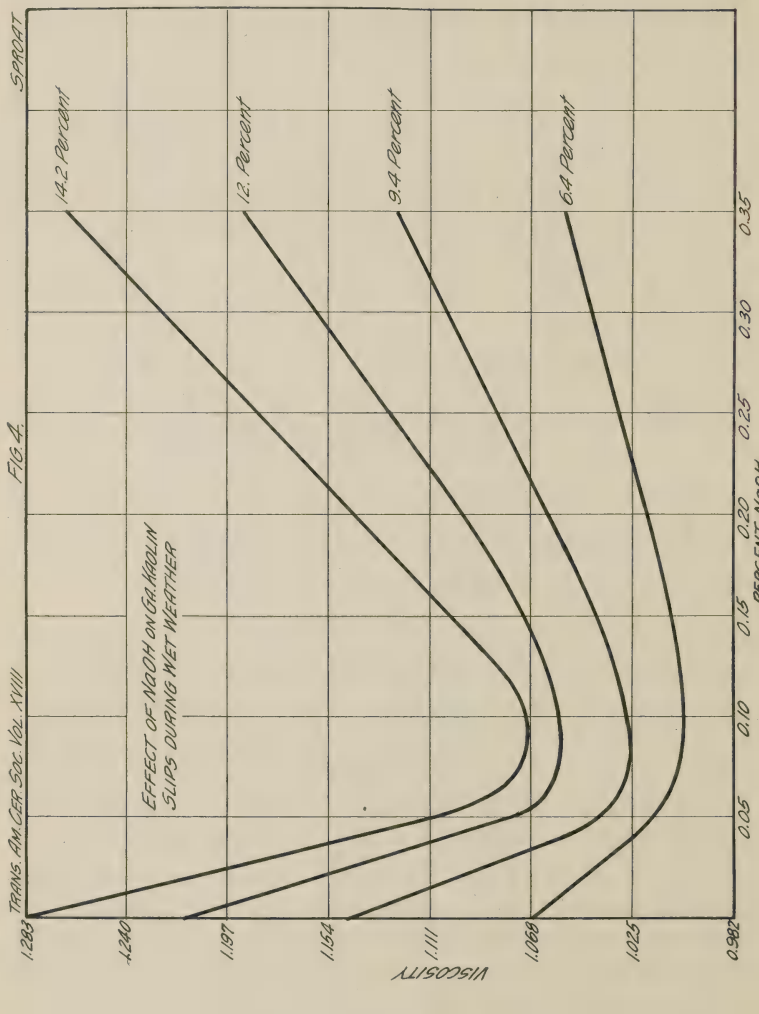
Alum. The results of tests made with slips containing a small percentage (0.03 percent) of alum are shown in Fig. 7. By comparing these curves with those of Fig. 3, it is seen that alum raises the initial viscosity, increases the amount of caustic soda necessary to produce minimum viscosity and decreases the degree of dispersion. Therefore the presence of a flocculating agent such as alum in the feed water is very objectionable to a successful application of the technical control of disperse systems in kaolin refining.

REFINING KAOLIN BY THE APPLICATION OF TECHNICAL CONTROL OF DISPERSE SYSTEMS

Sedimentation Process. From laboratory experience and from suggestions made by Ashley, Bleininger and others, it seems as if the best method of utilizing the influence of NaOH upon clay-water systems in kaolin refiining is as follows:

Sufficient NaOH to cause maximum deflocculation or minimum viscosity is added to the clay and water in the washer. The deflocculated slip is run through the settling troughs, screened and collected in the concentration tank, where it is allowed to stand for a sufficient time to allow the impurities to settle out. The slip is then syphoned into another tank and the flocculating agent added in order to coagulate the clay particles and make possible the concentration of the slip for filter pressing.

However, after making a number of tests in a small concrete tank six feet square and two feet deep, it was seen that any attempt to settle out the impurities in large tanks was not an economical process by any means, for the time required to settle out the impurities through great quantities of slip was comparatively long, being about two hours, and in the meantime a lot of the clay had also settled, and would have to be thrown away with the impurities. Besides, several inches of slip had to be


SPROAT
FIG 4.
TRANS. AM. CER. SOC. VOL. XVIII
EFFECT OF NaOH ON GA KAOLIN
SLIPS DURING WET WEATHER
14.2 Percent
12. Percent
9.4 Percent
6.4 Percent
VISCOSITY
1.283
1.240
1.197
1.154
1.111
1.068
1.025
0.982
0.05
0.10
0.15
0.20
0.25
0.30
0.35
PERCENT NaOH

left in the bottom of the settling tank after drawing off, as there was great liability of sucking up the impurities. Aside from the big loss of clay by use of such a method, it stops the continuity of the refining process and therefore adds to the operating costs.

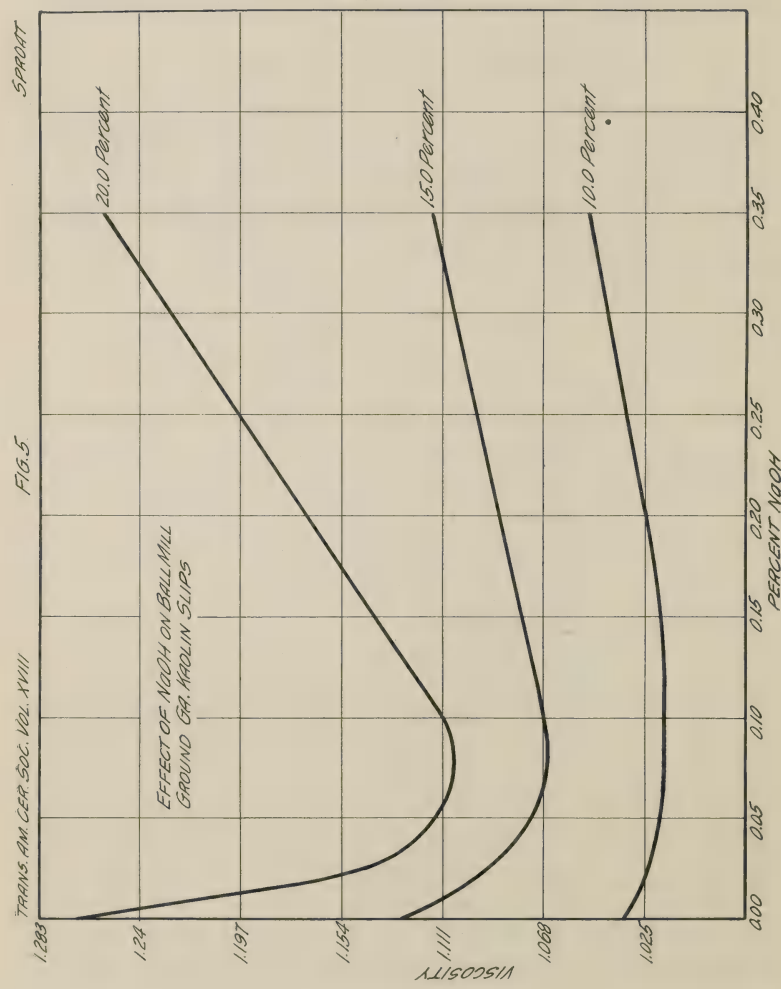
THE FLOTATION PROCESS

Experiments on settling out the impurities in floating troughs were next carried on. A new set of troughs being built for this purpose, and after a number of changes in width and length of the last trough, very good results were obtained. The complete floating process consists of eleven different operations which are as follows:

Storage and disintegration.
Feeding.
Blunging.
Dispersion.
Flotation.
Coagulation.
Screening.
Concentration.
Filter pressing.
Drying.
Crushing.

Storage and Disintegration. The clay must be broken up to lumps no larger than a man's fist, and most of the clay should be much finer. In order to disintegrate the clay in this manner, it is necessary to have a storage shed of at least 500 tons capacity, for during rainy weather the clay as mined from the pit is too sticky to be disintegrated.

All washing plants should have a storage shed for their crude clay regardless of the method of washing, for it has been shown that the capacity of the plant will vary greatly with the condition of the clay entering the washing unless it is dispersed with an alkali. However, few kaolin refining plants have a storage shed, therefore the writer wishes to emphasize the fact that the first thing to be considered in constructing a washing


SPROAT
FIG. 5
TRANS. AM. CER. SOC. VOL. XVIII
EFFECT OF NaOH ON BALL MILL GROUND GA. KAOLIN SLIPS
20.0 Percent
15.0 Percent
10.0 Percent
VISCOSITY
1.283
1.24
1.197
1.154
1.111
1.068
1.025
PERCENT NaOH
0.00
0.05
0.10
0.15
0.20
0.25
0.30
0.35
0.40

plant, if constant conditions are to be maintained with the resulting uniform product, is a good storage shed of sufficient capacity. The disintegrator should be of the corrugated roll type as this type works best with highly plastic clays.

It has been shown that the quantity of caustic soda required to produce maximum dispersion depends upon the percentage of clay present in the slip; therefore it is expedient to have a constant flow of clay into the washer. The best way to obtain this constant flow of clay is by the use of a clay feeder of the disc type. No feeder will deliver clay to a washer with any uniformity unless it has been previously crushed into small lumps. Hence the necessity of using a disintegrator.

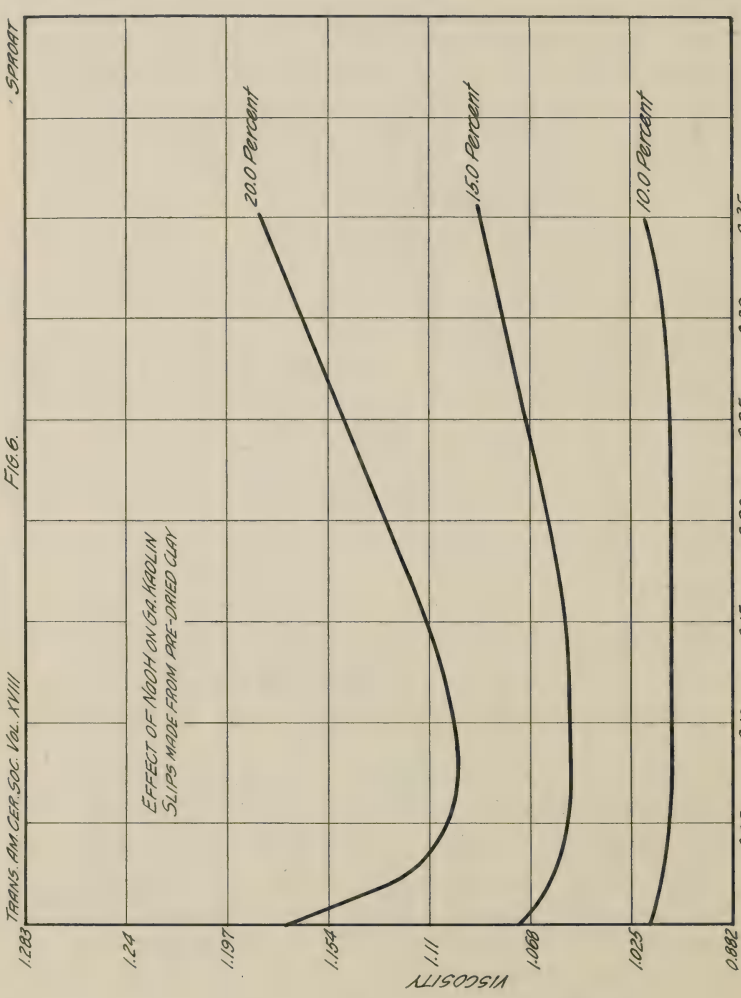
Blunging and Dispersion. These two steps in the process will be discussed together as they are very closely related.

The blunging of the clay, water and alkali is one of the most important steps in this washing process, and too much emphasis cannot be laid upon this operation. Double blunging or washing is necessary to obtain complete dispersion, and in the case of some very tough and tenacious clays which resist blunging to a great degree, it will be necessary to use three washers in order to get complete separation of the clay particles and impurities.

It is advisable to add caustic soda to the clay-water slip in the second blunger, for if for some reason the flow of clay is increased or decreased, the clay content of the slip can be calculated from viscosity tests on the slip leaving the first washer and the flow of standard NaOH solution regulated as to produce maximum dispersion under the changed flow of clay.

Addition of NaOH. A standard solution of caustic soda is made up and this solution is run into the second washer at a constant rate of about 10 cc. per second which is obtained by allowing the solution to run through an efflux tube of $\frac{1}{8}$ in. diameter under a constant head. The figures given above for the rate of flow of standard caustic soda solution and the size of the main efflux tube are somewhat arbitrary and can be changed to suit different conditions.

The strength of the NaOH solution will depend on both the rate at which it is allowed to flow into the washer and the


SPARGAT
FIG. 6.
TRANS. AM. CER. SOC. VOL. XVIII
EFFECT OF NaOH ON GA. KAOLIN
SLIPS MADE FROM PRE-DRIED CLAY
20.0 Percent
15.0 Percent
10.0 Percent
VISCOSITY
1.283
1.24
1.197
1.154
1.11
1.068
1.025
0.982
0.05
0.10
0.15
0.20
0.25
0.30
0.35
PERCENT NaOH

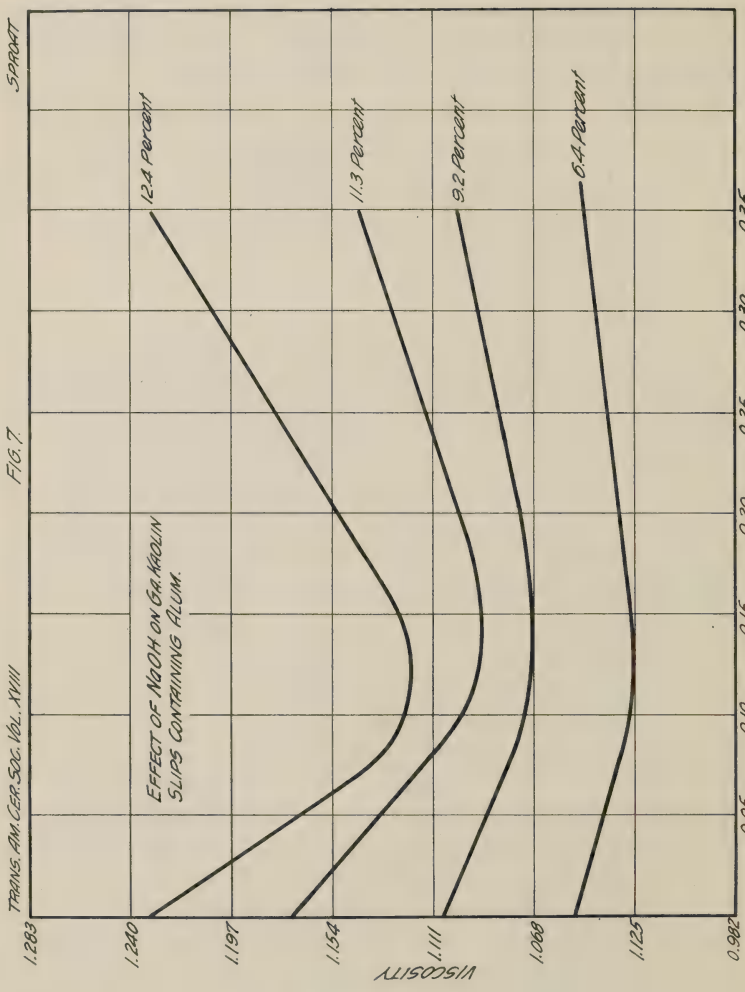
rate at which the clay is fed to the washer. For example: If five tons of clay are being fed to the washer every hour then 7.5 pounds (5 x 2000 x 0.00075) of NaOH will have to be added every hour to produce complete dispersion. If the rate of flow of the solution is 10 cc. per second, every cc. of standard solution will have to contain .094 grams of NaOH

$$\frac{7.5 \times 454.5}{3600 \times 10} = .094$$

There the normality of the solution is 2.35 N.-

$$\frac{.094 \times 1000}{40} = 2.35$$

Flotation. After thorough blunging and complete dispersion the slip is run through the floating troughs. The floating troughs used by the writer with good success do not differ from the ordinary mica troughs used at present in most washing plants, except that a long fan-shaped trough is added to the end of the other troughs. This fan-shaped trough should be at least 125 feet long, 2 feet wide at the inlet, while the width at the discharge end should be such as to produce a depth of slip of not over 1 inch. This fan-shaped trough must be constructed with great care for it must be perfectly level both ways and the sides of the trough very smooth so as not to cause too great a resistance to the flow of slip and a coagulation of the clay particles along the sides, and the resulting faster flow of the slip through the center of the trough. The same is true of the bottom of the trough except it does not want to be smooth but uniform in texture. If the bottom is made perfectly smooth the slip will have a tendency to carry the impurities along by sliding them along on bottom of trough. So the bottom must be rough in texture, but uniform; if not uniform the clay will have a tendency to coagulate around the spots of most resistance and therefore cause the slip to flow too fast in other portions of the trough. The best material for constructing these floating troughs is concrete, and by proper sizing of the aggregate and trowling the required surfaces can be obtained.


TRANS. AM. CER. SOC. VOL. XVIII
FIG. 7.
SPRAGUE
EFFECT OF NaOH ON GA. KAOLIN SLIPS CONTAINING ALUM.
12.4 PERCENT
11.3 PERCENT
9.2 PERCENT
6.4 PERCENT
VISCOSITY
1.283
1.240
1.197
1.154
1.111
1.068
1.025
0.982
0.05
0.10
0.15
0.20
0.25
0.30
0.35
PERCENT NaOH

There will be far less tendency of the clay particles to coagulate around spots of greater resistance if the slip is agitated before entering the last trough. This can be easily done by having the fan-shaped trough lower than the others and then running the slip over rifflers as it passes from the narrow troughs into the fan-shaped one.

Coagulation. As the slip comes from the floating troughs it is still in a state of maximum deflocculation and therefore could not be concentrated as the exceedingly fine particles would not settle even after days of settling. Furthermore, any attempts at filter pressing the slip as it comes from the floating troughs would be futile; for aside from the fact that the quantity of water to be removed would be so great that the process would not be an economical one, the alkaline condition of the slip with the resulting dispersion of the clay particles would make it almost impossible to filter press this slip. The exceedingly fine particles would tend to clog the filter cloth and what water did run then would be very turbid. Therefore it is necessary to add some flocculating or coagulating agent. Alum is usually used for this purpose. But it has been shown that small additions are exceedingly active in causing coagulation of the clay grains, therefore in cases where washing plants are using the water from the concentration tanks and filter presses over and over again, alum can not be used in connection with this process, as very small quantities (0.02 percent or less) present in the feed water will decrease the degree of dispersion upon addition of NaOH and also increase the amount of NaOH necessary to cause maximum dispersion. If just sufficient sulfuric acid is added to neutralize the caustic soda, the slip is brought back to normal state.

The strength of the sulfuric acid solution should be the same as the NaOH solution and added to the slip in the same manner. Under these conditions perfect neutralization will always occur, and there will not be an excess of NaOH left to cause trouble in settling and filter pressing, or an excess of H_2SO_4 left to cause trouble in the blunging and dispersion. The H_2SO_4 solution is added to the slip at the point where it leaves the floating trough. After the addition of the H_2SO_4

the slip is run through a long narrow trough (50 ft. by 1 ft, by 1 ft) containing a number of riffles for mixing the H_2SO_4 solution with the alkaline slip. This trough should have at least a drop of 1 foot in the 50 feet in order to cause a rapid flow.

Screening. After the slip becomes neutral upon the addition of H_2SO_4, it is advisable to run it through a rotary screen of about 120 mesh. This screen will remove any chips of wood or other foreign material that floats on the top of the slip, and it also acts as an agitator in case some of the H_2SO_4 had not completely neutralized the NaOH. However care should be taken that practically all of the H_2SO_4 has combined with the NaOH before reaching the screen, as any excess of H_2SO_4 will act upon the screen cloth, thereby reducing its life.

Concentration. The concentration of the slip is accomplished by running the neutral slip into large concrete tanks 75 feet long, 25 feet wide and 5 feet deep where it is allowed to stand for a sufficient period for the clay to be precipitated and the supernatant liquid to be drawn off, the time of settling governing the thickness of the slip. However, after about 48 hours the amount of additional precipitation is exceedingly small, and in general 24 hours is sufficient for the concentration of the slip. This concentrated slip is then pumped into the filter presses.

Filter Pressing. Under present conditions of kaolin refining the filter-pressing of the thickened clay slip has given more trouble than all the rest of the washing operations. The troubles have been numerous and varied and have been due partly to the type of filter-press, poor or clogged filter clothes, too thin a slip, condition of clay particles, dispersed or coagulated, etc. In view of these facts numerous attempts have been made to devise other methods of drying the clay, but to the writer's knowledge none have proved more efficient than the filter-press. The osmose purification process used in England [6] eliminates the filter-press, but it is a question if this apparatus would prove more efficient in this country.

Bleininger [7] says: "If the electrical continuous separation can replace the filter-press economically its use would be justi-

[6] *Trans. Eng. Cer. Soc.* 13, 35-48.
[7] Bleininger, page 51, *Tech. Paper 51*, Bureau of Standards.

fied. To a large extent it would be a matter of the comparative economy of the electrical endosmose apparatus and the filter-press. If the latter should be improved as has been done in the so-called clamshell press, it is quite possible that the economic advantage might remain with the filter-press."

The drying of the filter-press cakes is best accomplished in a tunnel dryer heated by exhaust or live steam. The filter-press cakes after being thoroughly dried are crushed into small pieces in a set of rolls.

Extra Cost of the Colloidal Chemical Process. The extra cost of the caustic soda-sulfuric acid process of refining kaolins over present-day methods may be divided into three groups: mechanical, chemical and chemist, all costs being figured on the basis of 50 tons per day.

The extra equipment necessary for the successful operation of this colloidal chemical process is: One disc clay feeder, one washer, extra floating trough and two 150-gallon tanks. The cost of operating and maintaining this extra equipment will be about $0.12 per ton.

The present (March, 1916) price of caustic soda is $0.03 per pound and that of sulfuric acid $0.025 per pound. Therefore the cost of chemicals will be $0.0825 per ton.

The successful operation of this process will depend upon the care taken with the collodial chemical end. It will therefore be necessary to have a ceramic chemist, one familiar with colloidal chemistry, on the job at all times. The services of such a chemist will cost at least $0.15 a ton.

The extra total cost will therefore be about $0.35 a ton. However, it must be remembered in comparing the cost of the two methods that the caustic soda-sulfuric acid process increases the capacity of the plan. For example, the slip of the maximum permissable viscosity of 1.094 after complete dispersion contains 17 percent of clay, while the slip that is not dispersed contains 10.8 percent during dry weather and 7.6 percent during wet weather. Taking the average percentage of clay, 7.64 percent, under the present dry methods, the increase in capacity will be 123 percent. Therefore this increased capacity will more than balance the increased cost of production.

SUMMARY

The application of colloidal chemical principles in kaolin refining on a commercial scale is thoroughly practical and the product obtained has a greater degree of purity which is not possible under present methods of refining. In order to utilize the $NaOH$-H_2SO_4 process it must be remembered that:

1st. The clay and water must be fed into the washer in a uniform rate.

2d. Double blunging or washing is necessary. Without thorough blunging the process is doomed to failure.

3d. The caustic soda must be added in definite quantities, the amount depending upon the character of the clay and the quantity being fed to the washer.

4th. A constant viscosity of about 1.068 must be maintained at the head of the floating troughs in order to obtain a uniform product. The viscosity should never exceed 1.094.

5th. It is advisable to agitate the slip before it is allowed to run through the fan-shaped trough.

6th. The last floating trough should be fan-shaped, at least 125 feet long, 2 feet wide at inlet, 7 feet wide at discharge end, and perfectly level both ways. The sides of this trough should be very smooth while the bottom comparatively rough but uniform in texture.

7th. Only sufficient sulfuric acid to just neutralize the caustic soda should be added.

8th. It is absolutely necessary to have a ceramic chemist, one familiar with clay disperse systems, on the job at all times, for it is impossible to make the process fool-proof.

PART II. THE UTILIZATION OF THE TREATED GEORGIA KAOLIN

Introduction. Having worked out an improved process of kaolin refining the question, "Will the treated clay be sufficiently improved so that it can be utilized in the manufacture of the finer grades of ceramic wares by substituting it for the English china clay?" at once arises. In order to answer this question several tons of clay were refined by the $NaOH$-H_2SO_4

process at the plant of the Georgia Kaolin Company, to be used for test purposes. Owing to the fact that during the refining of this clay the flow of clay into the washer was not uniform because a clay feeder had not been installed, and the amount of clay had to be approximately determined by viscosity tests taken every five minutes, it was not as pure as would be obtainable under standard conditions. However, upon making a number of settling tests it was thought that sufficient improvement had been made to warrant some preliminary tests on the burned color of the clay.

Preliminary Tests. The average linear shrinkage of the washed Georgia, treated Georgia and English china clays are given in Table III. The shrinkage was determined by measuring the dry length and burned length of a 5 cm. mark placed upon 10 cm. disks in the green state. These disks were made by pressing the plastic clay into plaster molds. The disks were dried at 110° C. and the clay lengths measured, then fired in saggers at cone 10 in the kilns of the Mayer China Company, and the burning shrinkage determined.

TABLE III

KAOLINS	DRYING SHRINKAGE	BURNING SHRINKAGE
	percent	*percent*
English china clay	2.32	4.48
Washed Georgia kaolin	4.38	8.12
Treated Georgia kaolin	3.24	8.16

From Table III it is seen that the $NaOH$-H_2SO_4 process decreases the drying shrinkage about 25 percent, but the burning shrinkage is about the same, probably a slight increase.

The preliminary tests on the use of the treated Georgia kaolin in white-ware body mixes were made in the ceramic laboratories of the Ohio State University. It was the purpose of these tests to determine how much improvement in burning color the treated clay would show over that of the washed Georgia kaolin when made up into glazed white ware bodies,

and also the approximate percentage of treated Georgia that could be substituted for the English china clay in a white ware body without impairing the color of the glazed body.

The commercial materials used in this investigation are as follows:

Feldspar — Canadian feldspar — Pennsylvania Feldspar Company.

Flint — Pulverized quartz sand — Ohio Silica Company.

Florida kaolin — Edgar's, Florida.

English China — Moore and Munger's M. W. M. No. 1.

Ball clay — Tennessee No. 9, Johnson & Porter Company.

Washed Georgia clay — Georgia Kaolin Company.

Treated Georgia clay — Georgia Kaolin Company.

Whiting — Gilders.

The analyses of these materials are given in Table IV.

TABLE IV

	FELDSPAR	FLINT	FLORIDA KAOLIN	ENGLISH CHINA	BALL CLAY	WHITING
Silica, SiO_2	65.15	99.18	45.50	47.37	51.20	1.30
Alumina, Al_2O_3	18.65	0.20	39.30	37.85	32.10	2.14
Iron oxide, Fe_2O_3	0.10	0.06	0.05	0.31	0.73	0.10
Titanium oxide. TiO_2	0.00	0.01	0.00	0.04	1.40	0.00
Lime, CaO	0.18	0.01	0.00	0.00	0.13	54.40
Magnesia, MgO	0.00	0.01	Trace	Trace	0.00	0.00
Potash, K_2O	10.36	.....	0.42	1.40	0.69	0.00
Soda, Na_2O	4.31	.....	0.44	0.14	0.10	0.00
Ignition loss	0.32	0.11	14.20	12.60	13.75	0.00
Carbon dioxide	0.00	0.00	0.00	0.00	0.00	42.68
	99.87	99.58	99.91	99.71	100.10	100.62

The numbers and composition of the bodies which were compounded from the above white burning materials are given in Table V.

TABLE V

	NO. 1	NO. 2	NO. 3	NO. 4	NO. 5	NO. 6	NO. 7
	percent	*percent*	*percent*	*percent*	*percent*	*percent*	*percent*
Feldspar	15.0	15.0	15.0	15.0	15.0	15.0	15.0
Flint	38.0	38.0	38.0	38.0	38.0	38.0	38.0
Florida kaolin	7.0	7.0	7.0	7.0	7.0	7.0	7.0
Tenn. ball clay	3.0	3.0	3.0	3.0	3.0	3.0	3.0
Whiting	2.0	2.0	2.0	2.0	2.0	2.0	2.0
English china	35.0	23.0	12.0	0.0	23.0	12.0	0.0
Washed Georgia	0.0	12.0	23.0	35.0	0.0	0.0	0.0
Treated Georgia	0.0	0.0	0.0	0.0	12.0	23.0	35.0

The bodies consisting of 8,000 grams were weighed out and the weight checked by weighing the total batch. Each body was ground wet for five hours in a porcelain lined ball mill, sieved through a 120-mesh screen and divided into two portions, the one portion being used for casting small cups while the other portion was dried out to a plastic state in plaster molds lined with cheese cloth, and after wedging was used for jiggering small flower pots.

The bodies were made up into 48 trials each, 24 by casting and 24 by jiggering. These trials were first air dried and then the drying completed in a gas-fired dryer.

In order that the degree of vitrification of these trials would cover the range of vitrification of commercial white ware bodies from the high porosity of wall tile to the very low porosity of vitreous china, one-half of the trials were biscuited at cone 4 and the other half at cone 11.

All trials were gloss fired at cone 3. The glaze used had the following molecular formula:

$$\left.\begin{matrix} 0.25\ PbO \\ 0.15\ K_2O \\ 0.05\ Na_2O \\ 0.54\ CaO \\ 0.10\ ZnO \end{matrix}\right\} 0.25\ Al_2O_3 \left\{\begin{matrix} 2.0\ SiO_2 \\ 0.4\ B_2O_3 \end{matrix}\right.$$

Frit composition:

$$\left.\begin{matrix} 0.10\ Na_2O \\ 0.90\ CaO \end{matrix}\right\} 0.1\ Al_2O_3 \left\{\begin{matrix} 2.0\ SiO_2 \\ 0.4\ B_2O_3 \end{matrix}\right.$$

The glaze was applied in a thick coat to one-half of the trials by dipping, and in a thin coat to the other half by spraying.

Results. Cone 12 biscuit — Cone 3 gloss —

TREATED VS. WASHED GEORGIA KAOLIN.

No black specks were observed in bodies Nos. 5, 6 and 7 containing treated Georgia kaolin, while a number of specks appeared in bodies Nos. 2, 3 and 4, especially in body No. 4 containing 35 percent of washed Georgia. Besides the treated kaolin bodies had a better color.

TREATED VS. ENGLISH CHINA CLAY.

Body No. 5 had just as good a color as body No. 1, while body No. 6 had a slight creamy tinge and body No. 7 a decided cream color when compared with body No. 1 containing all English china clay.

Cone 4 Biscuit, Cone 3 gloss.

TREATED VS. WASHED GEORGIA CLAY.

A few black specks showed up in body No. 4, but the difference in color between the treated and washed kaolin bodies was only slight at this heat-treatment.

TREATED VS. ENGLISH CHINA CLAY.

Bodies Nos. 5 and 6 had just as good color as body No.1, but body No. 7 showed a very slight creamy color when compared with No. 1.

Glazed white ware bodies containing the treated Georgia kaolin have a whiter color than those containing the washed Georgia. Treated Georgia kaolin can be substituted for at least 40 percent of the English china in vitreous china, and for all

the English china in wall tile bodies without affecting the color of the product.

These preliminary tests gave indications of the possible commercial uses of the treated Georgia clay and justified further tests on substituting it for English china clay. In order that these tests might be made under commercial conditions of white ware manufacture and the results practical, the Bureau of Mines secured the co-operation of the Beaver Falls Art Tile Company, The Mayer China Company and the Mosaic Tile Company.

Vitreous China. The commercial tests on the use of treated Georgia kaolin as a substitute for English china clay in vitreous china bodies were made at the Mayer China Company, Beaver Falls, Pa. The main object of these tests was to obtain if possible a practical vitreous china body composed of all American materials.

The materials used in this investigation, with the exception of the treated Georgia kaolin, were taken from the stock bins of The Mayer China Company. The following materials were used:

Feldspar — Eureka Flint and Spar Company.
Flint — Ohio Silica Company.
Florida kaolin — Edgar Plastic Kaolin Company.
English china clay — Moore and Munger's M. G. R. No. 2.
Ball clay — Moore and Munger's English ball clay.
Treated Georgia — Georgia Kaolin Company.

The body numbers and the percentage composition of each member of the three series comprising this investigation are given in Table VI.

TABLE VI

MATERIALS	SERIES A CONE 10 BISCUIT					
	20	21	22	23	24	25
Feldspar	16.0	16.0	16.0	16.0	16.0	16.0
Flint	37.5	37.5	37.5	37.5	37.5	37.5
Ball clay	7.5	7.5	7.5	7.5	7.5	7.5
Florida kaolin	8.0	8.0	8.0	8.0	8.0	8.0
English china clay	29.0	24.2	20.3	18.4	14.5	18.4
Treated Georgia kaolin	.....	5.8	8.7	10.6	14.5	.....
Washed Georgia kaolin	.....	.....	.....	.....	.....	10.6
Whiting	2.0	2.0	2.0	2.0	2.0	2.0
Magnesite	.....	.....	.....	.....	.....	.....
Percent total shrinkage	11.46	12.00	12.30	12.54	12.78	12.74
Percent drying shrinkage	2.42	2.60	2.78	3.00	3.17	3.16
Percent burning shrinkage	9.06	9.40	9.52	9.54	9.61	9.58
Percent absorption	0.05	0.41	1.00	1.08	1.30	1.02
Translucency in cm	0.65	.....	.....	.....	.....	.....

TABLE VI—Concluded

MATERIALS	SERIES B CONE 10 BISCUIT				SERIES C CONE 10 BISCUIT					
	26	27	28	29	29B	30	31	32	33	34
Feldspar	17.0	18.0	16.0	16.0	16.0	16.0	16.0	16.0	17.0	16.0
Flint	37.5	37.5	37.5	37.5	37.5	37.5	37.5	37.5	37.5	37.5
Ball clay	7.5	7.5	7.5	7.5	7.5	3.5	.....	.....	.....	.....
Florida kaolin	8.0	8.0	8.0	8.0	8.0	8.0	8.0	8.0	8.0	8.0
English china clay	16.0	15.0	14.5	13.5	13.5	17.5	19.5	17.5	13.5	19.5
Treated Georgia kaolin	12.0	12.0	13.5	13.5	13.5	13.5	15.0	17.0	20.0	15.0
Washed Georgia kaolin	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....
Whiting	2.0	2.0	3.0	4.0	4.0	4.0	4.0	3.0	2.0	2.0
Magnesite	.....	.....	.....	.....	.....	.....	.....	1.0	2.0	2.0
Percent total shrinkage	12.60	12.96	12.64	13.34	12.70	12.06	11.34	13.60	13.40	12.84
Percent drying shrinkage	2.84	3.00	3.20	3.16	3.08	2.24	2.50	3.14	3.22	2.58
Percent burning shrinkage	9.76	9.96	9.44	10.18	9.72	9.82	8.84	10.46	10.18	10.26
Percent absorption	0.26	0.03	0.60	0.02	0.16	1.19	2.26	1.05	0.76	1.04
Translucency in cm	0.61	0.64	0.61	0.68	0.40	0.45	0.46	0.51	0.53	0.54

The batch weights were determined from the percentage composition and figured to the basis of 8000 grams batch for each body. After weighing and checking the weight by weighing the total batch 1.2 grams of $CoSO_4$ were added for a stain, and the bodies blunged in the ordinary wet way in porcelain lined ball mills. Series "A" was ground for only five hours, but after the bodies of this series were biscuited it was found that the blunging was insufficient to give the desired uniform texture to the body; therefore the bodies of series "B" and "C" were blunged for 20 hours. After thoroughly grinding, the bodies were passed through a 120-mesh sieve and dried out to a plastic condition in plaster molds lined with cheese cloth. As soon as the bodies were sufficiently dry they were wedged on a marble slab, and then each body was made up into ten discs 10 cm. in diameter for shrinkage and absorption tests, and twelve 5-inch plates for color comparisons. Twenty-four wedge-shaped trials for transluscency tests were also made from each body of series "B" and "C". These trials were allowed to dry for about 12 hours and then settled.

The finished trials were placed in saggers in the ordinary commercial way, and placed in the second ring of the biscuit kiln. Series "A" and "B" were burned to cone 10, but Series "C" only received a heat-treatment of cone 9 due to bad heat distribution in the kiln in which the trials of this series were burned.

The glaze used had the following molecular formula:

$$\left.\begin{matrix} 0.058\ K_2O \\ 0.207\ Na_2O \\ 0.530\ CaO \\ 0.205\ PbO \end{matrix}\right\}\ 0.275\ Al_2O_3\ \left\{\begin{matrix} 2.540\ SiO_2 \\ 0.414\ B_2O_3 \end{matrix}\right.$$

Molecular formula of frit:

$$\left.\begin{matrix} 0.272\ Na_2O \\ 0.031\ K_2O \\ 0.697\ CaO \end{matrix}\right\}\ 0.272\ Al_2O_3\ \left\{\begin{matrix} 2.550\ SiO_2 \\ 0.544\ B_2O_3 \end{matrix}\right.$$

Eight kilograms of the above glaze were ground for sixty hours in a porcelain lined ball mill, passed through a 120-mesh

sieve and flocculated or thickened by the addition of 0.13 percent $CaCl_2$ (percent of dry weight). The thickened glaze was then applied to the biscuited plates by dipping in the ordinary commercial way.

The glazed trials were gloss fired at cone 1 in the glost kilns of the Mayer China Company.

Results. The physical and pyro-physical properties of the different bodies in this investigation will be compared with those of body No. 20, the composition of which was given to the writer by Mr. Ernest Mayer as a practical vitreous china body. The physical and pyro-physical properties that were studied are as follows: Working properties, drying-shrinkage, burning-shrinkage, absorption, transluscency and the color.

The working properties of the bodies were studied from the ease with which the ware was formed in the jigger without flawing in some way.

The drying and total shrinkage was determined by measuring the dry and burned lengths of 5 cm. marks placed on the 10 cm. discs in the green state. The drying and total shrinkage data given in Table VI is expressed in percent of green length. The burning shrinkage was calculated by subtracting the drying shrinkage from the total shrinkage.

The absorption data was obtained by weighing the dry discs and then soaking them in distilled water for 48 hours and again weighing. The average absorptions given in Table VI are expressed in percentages of dry weight.

The transluscency was determined by measuring the thickness of the wedge-shaped trial through which the light passed. The average transluscencies are given in cm. in Table VI.

Series "A". The purpose of this series was to study the direct substitution of treated Georgia kaolin for English china clay in a practical vitreous china body.

As the treated Georgia clay was increased the working properties of the body were improved, the drying and burning shrinkage and the absorption were increased. This decreased vitrification of the Geogia kaolin bodies is not due to the difference in fineness of grain of the English and Georgia clays, as is commonly thought, for the Georgia kaolin is much finer

grained than the English china clay, but is probably due to the low alkali content of the Georgia kaolins.

The color of bodies Nos. 21, 22 and 23 were just as good as that of body No. 20, while body No. 24 showed a very slight tendency toward a cream color, therefore only about 50 percent of the English china clay in an ordinary vitreous china body can be replaced by treated Georgia kaolin before the color of the glazed product is impaired.

Series "B". This series was designed to obtain a body of low absorption and still contain the maximum percentage of treated Georgia kaolin. To do this the feldspar (bodies 26 and 27) and the whiting (bodies 28 and 29) were increased.

The working properties of the four bodies of this series were very good. The burning shrinkage was increased with the resulting decreased absorption and increased transluscency. The color of all the bodies of this series was just as good as that of body No. 20. Body No. 29 was a good vitreous china body of high transluscency.

Series "C". The purpose of this series was to study the effect of substituting treated Georgia kaolin for ball clay and magnesite for a portion of the whiting. Owing to the decreased biscuited heat-treatment the bodies of this series had an abnormal high absorption and low transluscency, as may be seen by comparing the data for bodies 29 and 29-B, Table VI. These two bodies had the same composition but were biscuited at different heat-treatments.

Substituting treated Georgia for ball clay decreased the drying and burning shrinkage and increased the absorption. The transluscency was increased regardless of the decrease in degree of vitrification.

Magnesite had a greater fluxing action than whiting, as is seen by comparing the absorption of body No. 31 with No. 34.

A direct substitution of Georgia clay for ball clay made the body too short and therefore greater amounts of Georgia kaolin must be used to produce a practical vitreous body. Bodies 32 and 33 containing 17.0 percent and 20 percent respectively, had good plasticity, and worked well in the jigger, while bodies Nos. 30 and 31 were too short.

The color of bodies Nos. 31, 32, 33 and 34 of this series had a much better color than body No. 20 or 29 which contained ball clay. Bodies 33 and 34 had the best color of the series which was evidently due to the substitution of magnesite for 50 percent of the whiting.

Summary. A commercial vitreous china body could not be obtained by the direct substitution of Georgia kaolin for English china clay, but the fluxes had to be increased to obtain the desired degree of vitrification.

The substitution of treated Georgia for all the ball clay and 50 percent of the English china clay produced a body (No. 33) of good working qualities and as good a color as the whitest commercial ware and a far better color than most vitreous china ware on the market.

As far as color is concerned more than 20 percent of treated Georgia kaolin can be used in bodies containing no ball clay, but the writer is of the opinion that if greater quantities were added the biscuited heat-treatment would have to be increased in order to secure the desired vitrification, for an increase in percentage of fluxes over that in body No. 33 would reduce the toughness of the ware, and as this is an important factor in high-grade vitreous china or hotel china, 20 percent of treated Georgia is about the maximum amount that can be used in this class of ware. Therefore in order to produce a vitreous china body containing all American materials, it will be necessary to substitute for the 13.5 percent English china clay in body No. 33 a domestic kaolin that is as easily vitrified as the English china clay, and having as good a burning color.

Wall Tile. The commercial tests on the substitution of treated Georgia clay for English china clay in wall tile were made at the Beaver Falls Art Tile Company, Beaver Falls, Pa., and the Mosaic Tile Company, Zanesville, Ohio. As the tests at the Beaver Falls Art Tile Company were of a preliminary nature they will be discussed first. The purpose of these tests was to determine the maximum amount of Georgia clay that could be made in a commercial wall tile. From the preliminary tests made at the Ohio State University it was seen that at least comparatively large amounts could be used in bodies of high ab-

sorption before the color was affected, but what effect large amounts would have on other pyro-physical properties of wall tile had to be determined in these preliminary tests at the Beaver Falls Art Tile Company.

The materials used in this investigation were as follows:

English china clay — Moore and Munger's M. W. M.
Ball clay — Kentucky No. 4.
Florida kaolin — Edgar Plastic Kaolin Company.
Flint — Pennsylvania Pulverizing Company.
Feldspar — Pennsylvania Feldspar Company.
Treated Georgia kaolin — Georgia Kaolin Company.

As these tests were to form the basis of larger commercial tests at the Mosaic Tile Company, it was thought advisable to determine the fineness of grain of the flint and feldspar, in order that the shrinkage and absorption could be controlled upon changing to a different feldspar and flint. The mechanical analyses were made on a Schultze elutriation apparatus[8] with an overflow of 80 cc. per minute, and are given in Table VII.

TABLE VII

SEPARATES	FELDSPAR	FLINT
Residue on 120 Mesh Sieve. Average diam. of grain .186 mm	9.46	0.60
Residue on 150 Mesh Sieve. Average diam. of grain .157 mm	0.90	0.42
Residue on 200 Mesh Sieve. Average diam. of grain .110 mm	7.50	1.56
Residue on 260 Mesh Sieve. Average diam. of grain .0765 mm	10.70	9.30
Residue on 330 Mesh Sieve. Average diam. of grain .0578 mm	9.36	4.92
Residue in Elutriator Jar No. 1. Average diam. of grain .034 mm	20.84	25.74
Residue in Elutriator Jar No. 2. Average diam. of grain .0187 mm	9.60	18.00
Residue in Elutriator Jar No. 3. Average diam. of grain .010 mm	10.80	19.06
Overflow from Jar No. 3	20.84	20.40

[8] Page 46, *Bulletin 53, Bureau of Mines.*

TABLE VIII

MATERIALS	SERIES A					SERIES B			SERIES C		
	10	11	12	13	14	15	16	17	18	19	20
	percent	*percent*	*percent*	*percent*	*percent*	*percent*	*percent*	*percent*	*percent*	*percent*	*percent*
Feldspar	15.0	15.0	15.0	15.0	15.0	13.0	12.0	11.0	12.0	12.0	12.0
Flint	40.0	40.0	40.0	40.0	40.0	40.0	40.0	40.0	40.0	40.0	40.0
Florida kaolin	10.0	10.0	10.0	10.0	10.0	11.0	11.0	12.0	....	....	....
Kentucky ball clay	15.0	15.0	15.0	15.0	15.0	16.0	16.0	15.0	15.0	15.0	....
English china clay	20.0	15.0	10.0	5.0	....	....	5.0	....	33.0	....	....
Treated Georgia clay	....	5.0	10.0	15.0	20.0	20.0	16.0	20.0	....	33.0	48.0
Whiting	....	....	....	....	....	....	....	1.0	....	....	....
Magnesite	....	....	....	....	....	....	....	1.0	....	....	....
Shrinkage	9.70	9.57	9.37	9.15	8.51	6.39	6.10	5.76	6.74	4.59	3.41
Absorption	6.75	7.52	7.98	8.70	9.65	12.44	12.65	12.27	10.02	13.86	17.70
Shade	D	D	D	D	D	B	B	B	C	C	A

The percentage composition of the bodies compounded from the above white burning materials is given in Table VIII.

Ten kilograms of each body were weighed out, and the weight checked by weighing the total batch. The bodies were blunged for five hours in a small blunger constructed from an ice cream freezer, the slip passed through a 120-mesh sieve and filter pressed in a small laboratory press. The press cakes were dried to bone dryness at 110° C. and then ground to pass 20-mesh sieve. 13 percent of H_2O was added to the ground bodies, these were thoroughly mixed by hand, and then reground in a small dust mill to pass 30-mesh. The dust was stored in stoneware jars over night.

The bodies were pressed into standard 6 in. by 3 in. tile on an ordinary hand screw press. In order to obtain uniform pressing, all bodies were pressed by the same experienced pressman. Thirty tile were pressed from each body which were divided into six sets of five tile each for distribution in different sections of the kiln.

The tile were set in saggers in the ordinary manner. Each sagger contained five tile of each body composition, and were distributed in the kiln in the following way.

A — Bottom of 1st ring, just above log wall.
B — Top of 1st ring.
C — Bottom of 2nd ring.
D — Top of 2nd ring.
E — Bottom of 3rd ring.
F — Top of 3rd ring.

A pat of cones containing cones 9, 10, 11 and 12 were placed in each sagger. The heat-treatment that the bodies in different sections of the kiln received is as follows:

KILN SECTION	CONE
A	13
B	11
C	11
D	10
E	9
G	12

The burn was made in an ordinary down-draft Rivers kiln. The bad heat distribution which is evident from the above table was due to a change in burners.

One tile of each body composition from each section of the biscuit kiln was glazed with the regular glaze used by the Beaver Falls Art Tile Company. The glaze was applied by dipping.

A study of the vitrification was made by the determination of average absorption and shrinkages. The absorption was determined by soaking the biscuited tiles in distilled water for 48 hours and expressing the amount of water absorbed by each tile in percentage of dry weight. The shrinkage was determined by measuring the burned length of tile and expressing the difference between the green length (length of die) and burned length in terms of percentage of green length. The different heat-treatments which the trials in different sections of the kiln received, had a decided effect on the absorption and shrinkage. This may be emphasized by the actual results of Body No. 18 given in Table IX.

TABLE IX

KILN SECTION	ABSORPTION	SHRINKAGE
	percent	*percent*
A	7.94	7.43
B	10.19	6.90
C	9.75	6.88
D	10.66	6.55
E	12.30	5.83
G	9.29	6.88

The average shrinkages and absorptions are given in Table VIII.

As there is no standard method of measuring the shade or color of a wall tile, the shade of the glazed trials was determined by comparison. The results are given in Table VIII, "A" being the whitest shade and "D" the darkest.

Results. Series "A"—The purpose of this series was to study the effect of substituting treated Georgia kaolin for English china clay. From the results tabulated in Table VIII it is seen that as the Georgia kaolin was increased, the vitrification

was decreased, but no change in color was noted. All the bodies of this series had too high a shrinkage and to low an absorption for practical wall tile, as most commercial wall tile have a shrinkage of about 4.5 percent and an absorption of 14.0 percent.

Series "B"—This series was designed to decrease the vitrification of members 13 and 14 of series A. From the data in Table VIII it is seen that the shrinkage was reduced and the absorption increased but not sufficient to approximate that of commercial wall tile. An improvement in color was noted; this being due to decreased vitrification. The introduction of 1 percent whiting and 1 percent of magnesite did not improve the color of the tile.

Series "C".—The purpose of this series was to further reduce the vitrification by substituting treated Georgia clay for Florida kaolin and ball clay. The results tabulated in Table VIII speak for themselves. However, the most interesting condition was found in bodies 18 and 19, when 33 percent of treated Georgia kaolin replaces 33 percent of English china clay without impairing the color of the glazed product. Another interesting fact was the improved color caused by substituting treated Georgia kaolin for ball clay (Body No. 20), and in spite of the fact that the Georgia kaolin composed 48 percent of the body composition. The shrinkage and absorption of body No. 19 approximated that of commercial wall tile and was therefore used as a basis for further investigation on wall tile bodies.

Tests at the Mosiac Tile Co. In the preliminary tests at the Beaver Falls Art Tile Co., the treated Georgia kaolin gave such good promise of being an ideal clay for wall tile manufacture that large commercial tests were thought advisable. These tests were carried on at the plant of the Mosaic Tile Company, Zanesville, Ohio.

The materials used in this investigation were as follows:
Flint — Pennsylvania Pulverizing Company.
Feldspar — Pennsylvania Pulverizing Company.
Florida kaolin — Edgars Plastic Kaolin Company.
Ball clay — Cooley Ball.
Treated Georgia clay — Georgia Kaolin Company.

TABLE X

MATERIALS	A		B									C
	43	44	45	46	47	48	49	50	51	52	53	54
Flint	40.0	40.0	40.0	40.0	40.0	40.0	40.0	40.0	37.5	35.0	35.0	35.0
Feldspar	13.0	.....	13.0	14.0	14.0	14.0	15.0	16.0	14.0	14.0	14.0	13.0
Cornwall stone	.....	14.0	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....
Florida kaolin	12.0	11.0	12.0	11.0	6.0	.....	.....	.....	.....	.....	.....	.....
Ball clay	.....	.....	.....	.....	5.0	11.0	5.0	.....	13.5	16.0	10.0	14.0
Treated Georgia clay	35.0	35.0	35.0	35.0	35.0	35.0	40.0	44.0	35.0	35.0	41.0	38.0
Percent shrinkage	3.00	3.00	5.28	5.44	5.92	6.40	6.56	7.20	6.88	7.85	7.52	4.67
Percent absorption	17.70	18.00	17.09	17.19	15.10	14.25	14.19	15.19	14.74	14.21	15.37	14.40
Breaking strength in lbs.	45.0	52.0	55.1	55.5	68.5	81.5	71.7	76.5	85.0	100.8	87.0	70.0
Relative shade	A	A	.....	.....	.....	.....	.....	.....	.....	.....	.....	D

The numbers and percentage composition of the bodies included in this investigation are given in Table X.

The bodies of series A and B consisting of 3000 pounds batch were prepared as follows: The total batch was blunged in a double blunger for one hour in the ordinary wet way, and then the slip was passed through a 120-mesh vibratory screen into an agitator from where it was pumped into the filter-presses. The filter press cakes were dried in a tunnel dryer heated by the waste heat from cooling kilns and then crushed into small pieces in a set of rolls. About 11 percent of water by weight was added to the crushed body, and after standing for several hours it was ground to a dust in an ordinary dust mill of the fan type. The dust was collected in a large storage bin from where it was taken to the presses.

The batch weight of the bodies of series B was only 8000 grams, and they were prepared as follows:

1. Bodies weighed and weight checked by weighing total batch.
2. Blunged in porcelain lined ball mills for one hour.
3. Sieved through 120-mesh screen.
4. Slip concentrated in plaster absorption bowls lined with muslin and finally dried completely in dryer.
5. Crushed to pass 20-mesh screen.
6. Tempered by the addition of 11 percent of water by weight.
7. Ground to pass 20-mesh screen.
8. Allowed to stand for several hours in covered jar.

The bodies were pressed into 6 in. by 3 in. and 6 in. by 6 in. tile on the power presses of the Mosaic Tile Co.

The tile after being allowed to dry for about four days were set in saggers and biscuited at cone 11. The biscuited tile were then glazed with the regular glaze used by the Mosaic Tile Co. and gloss fired at cone 02.

Results. The absorption, shrinkage and color data given in Table X were obtained in the same manner as in the preliminary tests. The breaking strengths given in this table were obtained by determining the weight that would break the biscuited tile

gripped between two steel plates. The weight was suspended 2½ inches from the point of support.

Series "A". The object of this series was to study the effect of feldspar vs. Cornwall stone in wall tile bodies. From the data in Table X it is seen that it takes 1 percent more Cornwall stone to produce the same degree of vitrification but the stone increases the strength of the body about 15 percent. The bodies of this series had too high an absorption and too low a shrinkage and breaking strength for good commercial wall tile.

Series "B". The purpose of this series was to obtain a body of higher breaking strength. By comparing body No. 45 of this series with body No. 43 of series A, it is seen that grinding a body in a ball mill instead of blunging increased the shrinkage but had very little effect on the absorption. The breaking strength was greatly increased by substituting ball clay for Florida kaolin, and by increasing the clay content at the expense of the flint a further increase in strength was obtained, reaching a maximum of 100.8 pounds in body No. 53. As none of the bodies of this series were gloss fired no relative shade is given for these bodies.

Series "C". A large commercial test was made on a body of the composition of No. 54, Table X. This body proved to be a good commercial wall tile body. The strength was 70 pounds which is low, due to the fact that the dust was too dry when pressed. The shade or color of the glazed trial was as good as some of the leading wall tile on the market.

Summary. Treated Georgia kaolin can be substituted for all the English china and a portion of the ball clay without impairing the color of the burned product.

Cornwall stone makes a stronger body than feldspar, but a feldspar wall-tile body can be made that is as strong as any commercial wall tile on the market, providing that the total clay content is at least 52 percent and 10 percent of this must be ball clay and the remainder treated Georgia kaolin.

The writer wishes to express his indebtedness for assistance in this work to Dr. Charles L. Parsons, Prof. Arthur S. Watts, and the co-operating firms.

FRITTED LEADLESS GLAZES FOR SANITARY WARE

BY CULLEN W. PARMELEE AND GEORGE A. WILLIAMS

The purpose of this investigation has been the study of the development of glazes of the fritted leadless type for the porcelain-like body used in the manufacture of sanitary ware. Such bodies are made of white burning clays, china clays and ball clays, flint and feldspar. They are biscuited and glazed at cones 7 to 9. The glazes commonly used on such wares are of the Bristol type with the addition of sufficient lead to obtain a better gloss and lower viscosity than is usually obtained with the true Bristol glaze. Glazes of the porcelain type may possibly be had for this purpose. In fact one of the authors has seen such, but the usual glaze of this sort does not give the qualities desired.

The advantages and the objections to the use of lead in glazes are too familiar to necessitate restating.

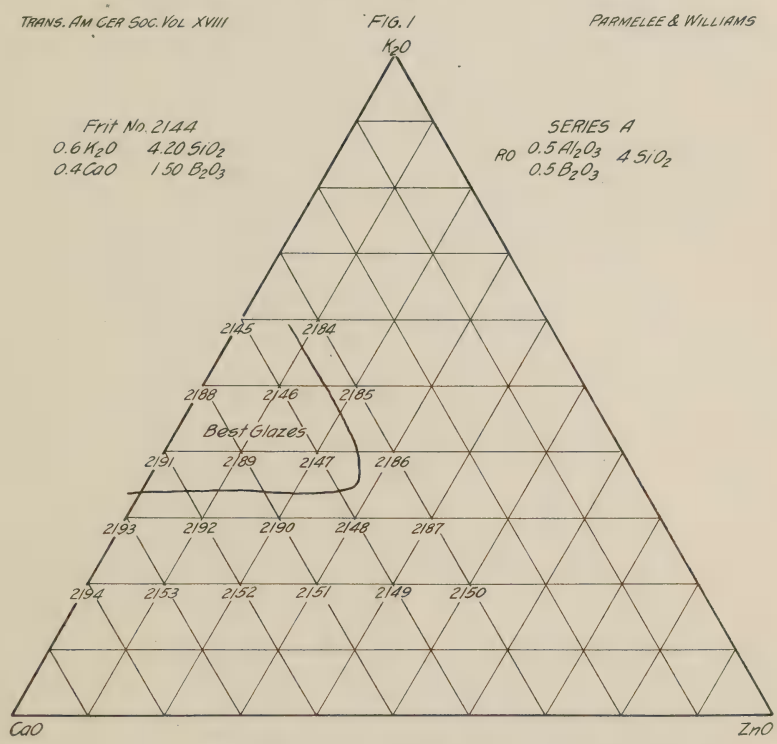
Glaze Materials. These are given in the accompanying tables of batch weights. No one frit would serve for all the glazes, therefore, several of different composition were used. The formulae and batches of the same are shown in the accompanying tables. We are aware, of course, that the composition of the frit and the amount used are important factors in the development of glazes of this type.

However, it was not feasible to attempt to use one frit for all glazes, consequently we have chosen to neglect this detail which, in itself, would require a great deal of experimentation. We offer these results as a basis upon which other work may properly rest. In view of the fact that the frits used are of varying composition, we may group the various series together according to the frits used.

Arrangement of Work. The experimental work was arranged according to the following plan:

Series A — This group of glazes has the same equivalents of alumina, boric oxide and silica. The R O members are varied as shown in Fig. 1. For this series one frit, No. 2144, was used throughout.

Series B — The alumina, boric oxide and silica equivalents are constant and the same as in Series A. The R O members are the same, but vary as shown in Fig. 2. This series differs

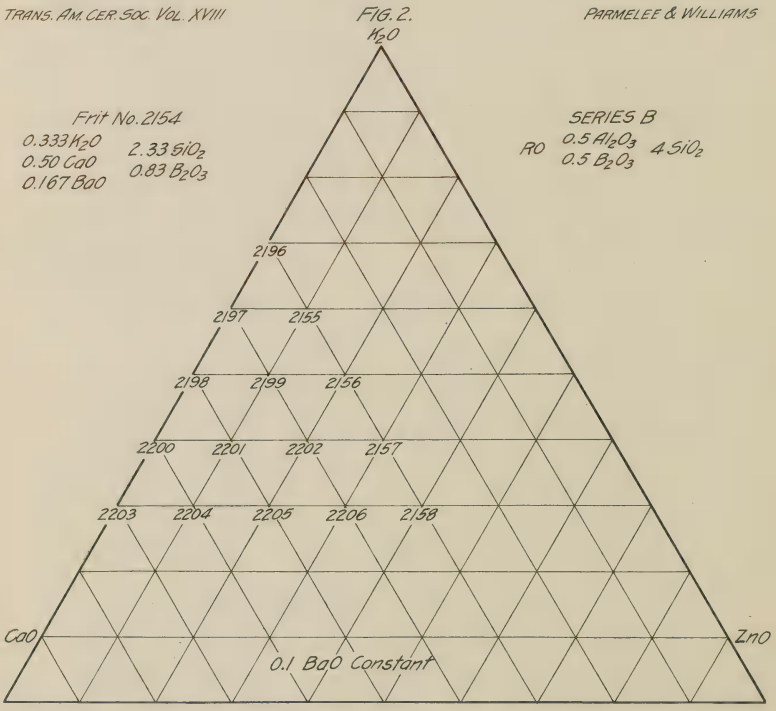


from Series A in that 0.1 equivalent of barium oxide was introduced as a constant, with the expectation that this ingredient would give greater fusibility and greater brilliancy to the glaze than could be had in Series A.

Series C — The glazes of this group have the same equivalents of alumina, boric oxide and silica, also the same as in Series A and B. The R O members vary as shown in Fig. 3.

The series differs from Series A in that the alkalies are equally divided between soda and potash.

Series D — The members of this group of glazes have the same equivalents of alumina and boric oxide and silica which are the same as in Series A, B and C. The R O members vary as shown in Fig. 4. This series is identical with Series C, excepting that the potash is introduced in the form of nitrate of potash in Series C and as pearl ash in Series D.

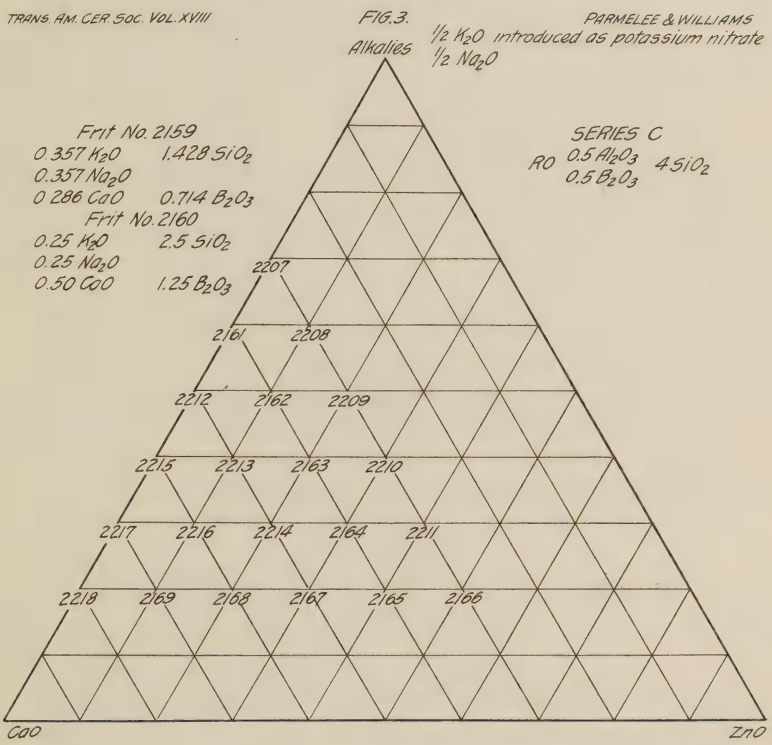


The Series A, B, C and D were arranged for the purpose of determining the most favorable R O compositions. From Series A we selected six glazes, Nos. 2145, 2146, 2147, 2188, 2189, 2191, which appeared to be in the area of the best glazes. These glazes were taken as the basis of the following series.

Series E — The R O members of this series were selected as described, and in all the alumina and silica remain constant, but

the boric oxide content varies from 0.5 equivalent to 0.1 equivalent. The purpose of this series was to confirm the results obtained in Series A, to afford a better opportunity for the selection of the best R O composition, and to compare the effects of varying the boric oxide content.

Series F — This series is based upon a glaze No. 2188, which was selected as one of the best of Series A and Series E.

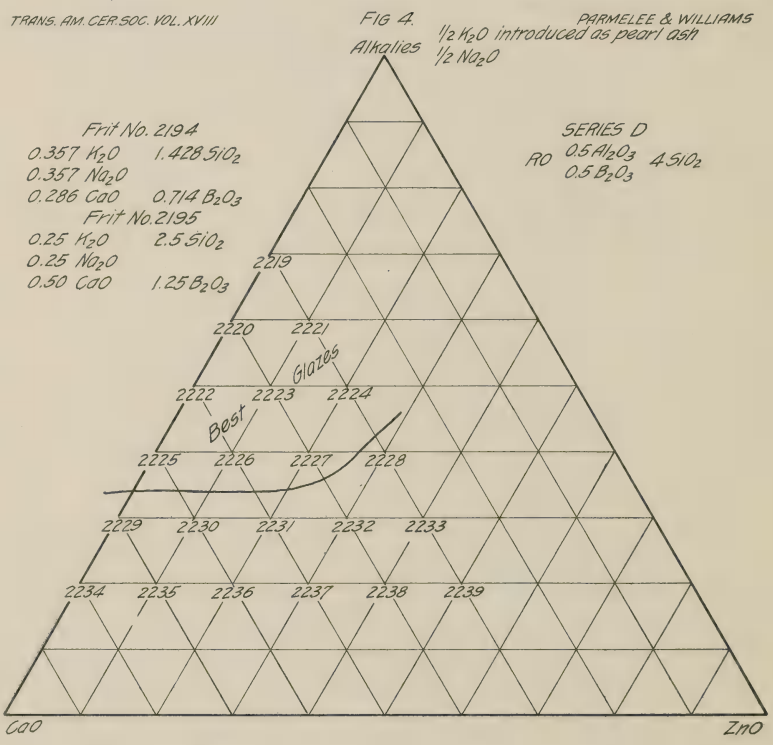


The R O members and the alumina remain constant. The variables are the silica, ranging from 3. equivalents to 7. equivalents, and the boric oxide which varies from 0.1 equivalent to 0.5 equivalent. The same frits are used in Series E and F.

Series G — This series has a constant R O, which is the same as in Series F, *i. e.*, like glaze No. 2188, and the silica is constant at 3 equivalents. The variables are alumina ranging

from 0.3 to 0.7 equivalents and boric oxide ranging from 0.1 to 0.5 equivalents.

Series H — This series has a constant R O which is the same as in Series F and Series G; namely, like glaze No. 2188 of Series A. The alumina and the boric oxide are variables of the same range as in Series G. The silica is constant at 4 equivalents.



Series I — This series is like Series G and H in having the same R O composition and the same variations in the alumina and boric oxide. The silica content is constant with 5. equivalents.

The Series F, G, H and I are, therefore, alike in being based upon a constant R O which is also the same as that of glaze No. 2188 of Series A and Series E. It will also be seen that certain members of Series F are to be found in Series G, H

and I, thus furnishing a check. These check glazes were found to be similar in general characteristics, although not exactly alike, which may be attributed to the differences in the frits used. Series G, H and I are comparable, because they are made with the same frits.

Body and Burning. Through the kindness of Trenton friends we were favored with the opportunity of trying the glazes in commercial kilns and on commercial bodies. Our laboratory burns were of about twenty-four hours duration, while the commercial kilns are burned for a considerably longer period, therefore, we found as might be expected some differences in the results. The results of our laboratory trials were in general confirmed. The essential difference being that larger numbers, *i. e.*, larger areas of good glazes were obtained in the commercial kilns. These glazes were applied upon biscuited tile made from commercial bodies The burns were made at cone 7

Frits. Table I shows the compositions of the frits, the series in which they were used and their deformation points. The latter were obtained by molding cones from the finely ground fused frits and determining the temperatures of deformation of the same in a Seger furnace, using a thermo-couple to record the temperatures.

TABLE I. COMPOSITION OF FRITS

NUMBER	NO. 2144	NO. 2154	NO. 2159	NO. 2160	NO. 2144*	NO. 2386	NO. 2499	NO. 2500
USED IN SERIES	A	B	C, D	C	E, F	E, F	G, H, I	G, H, I
Na_2O	....	....	0.357	0.25	0.60	0.60	0.667	0.667
K_2O	0.60	0.33	0.357	0.25	.....	.....	.....	.....
CaO	0.4	0.50	0.286	0.50	0.40	0.40	0.333	0.333
BaO	....	0.167	.....	.....	.....	.....	.....	.....
B_2O_3	1.50	0.83	0.714	1.25	1.50	0.30	0.83	0.167
SiO_2	4.20	2.33	1.428	2.50	4.20	4.20	2.0	2.0
Deformation Point	775° C.	770° C.	710° C.	740° C.	775° C.	885° C.	680° C.	740° C.

* The same frit was used in Series A.

RESULTS OF BURNS AT CONE 7

Series A. Crazing occurs only on sample No. 2184.

Opalescence. In the report on these experiments we apply this term to all appearances in the glaze of a cloudy or milky sort, whether associated with a play of color or not, since we have found in this investigation that these phenomena are evidently related. The trials showing opalescence are Nos. 2145, 2148, 2146, 2185, 2189, 2147. Those highest in potash show it most strongly.

Gloss. This increases with the increase in the zinc content.

Color. Those highest in calcium are the whitest.

Opacity increases as the calcium increases.

Texture. The trial with the highest zinc content shows a blistered surface. Those highest in calcium have a semi-glossy, wavy surface. Those highest in potash have a dull egg-shell surface.

Good glazes are found in the area bounded by Nos. 2145, 2147 and 2191. Number 2186 has a very good surface and more gloss than the others, but is near the boundary limiting the good area since No. 2187 has crawled.

Series B. Crazing — None.

Opalescence — None.

Gloss. The various glazes have about the same gloss which is fairly good. The influence upon the gloss of an increase in the zinc content is very slight. It apparently makes the glazes whiter.

Opacity increases as the zinc content increases.

Texture. Glazes with lowest content of alkali have a slightly wavy surface. Others show an egg-shell surface. The glaze highest in calcium has crawled badly.

Best glaze appears to be No. 2157.

Series C. Crazing appears on sample No. 2207.

Opalescence appears on No. 2207.

Gloss. Most of the glazes are glossy, especially as the zinc content increases. Those highest in calcium are duller.

Color. Those highest in alkali are not as good as the others.

Textures. Those highest in zinc have blistered.

Best glazes are Nos. 2214,2212 and 2146.

Series D. Crazing. Only No. 2219 has crazed, possibly No. 2220 also.

Gloss. The best gloss is found in those glazes highest in alkalies. Those having the highest zinc are also glossy, but have various surface defects. The other glazes are more or less dull depending upon the lime content.

Opalescence. Glazes No. 2220 and 2224 show opalescent areas. Glazes No. 2219, 2220 and 2221 show surface markings, apparently of a crystalline character.

Color. The color of the glazes is very good.

Texture. The glaze textures of this series are generally very good. Those glazes having the highest zinc content have surface defects such as blistering or crawling.

Best glazes. The best glazes are Nos. 2221, 2224 and 2226. The area of best glazes is bounded by the glazes No. 2219, 2225 and 2228.

Series E. R O 0.5 Al_2O_3 4 SiO_2 x B_2O_3

Equiv. of B_2O_3

0.5	0.4	0.3	0.2	0.1
2145	2387	2388	2389	2390
2146	2406	2407	2408	2409
2147	2391	2392	2393	2394
2189	2410	2411	2412	2413
2191	2395	2396	2397	2398
2188	2402	2403	2404	2405

The R O members are as of the glazes indicated in the first column to the left and remain constant along the horizontal lines.

The R O members vary as one reads the column vertically.

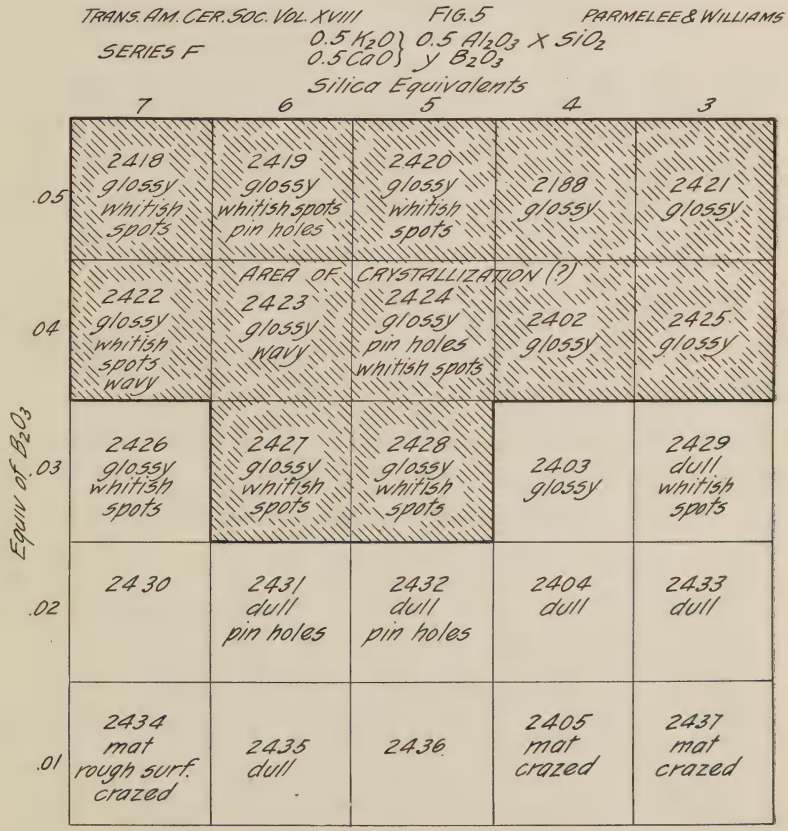
Crazing appears on glazes Nos. 2405, 2390.

Opalescence appears in glazes Nos. 2145, 2387, 2146, 2406 and 2147, also 2402 where thickly dipped.

Gloss. The gloss decreases with the decrease in the content of boric acid although the differences are slight. Glazes

without zinc, *i. e.* like Nos. 2146, 2188 and 2191 decrease in gloss much more markedly with decreasing zinc content.

Texture. Best texture is shown by glaze No. 2188, Nos. 2390 and 2405 are good mats. Most of the glazes show slight egg-shell texture.



Series F. Crazing is found on glazes Nos. 2434 and 2437, *i. e.* glazes having lowest silica.

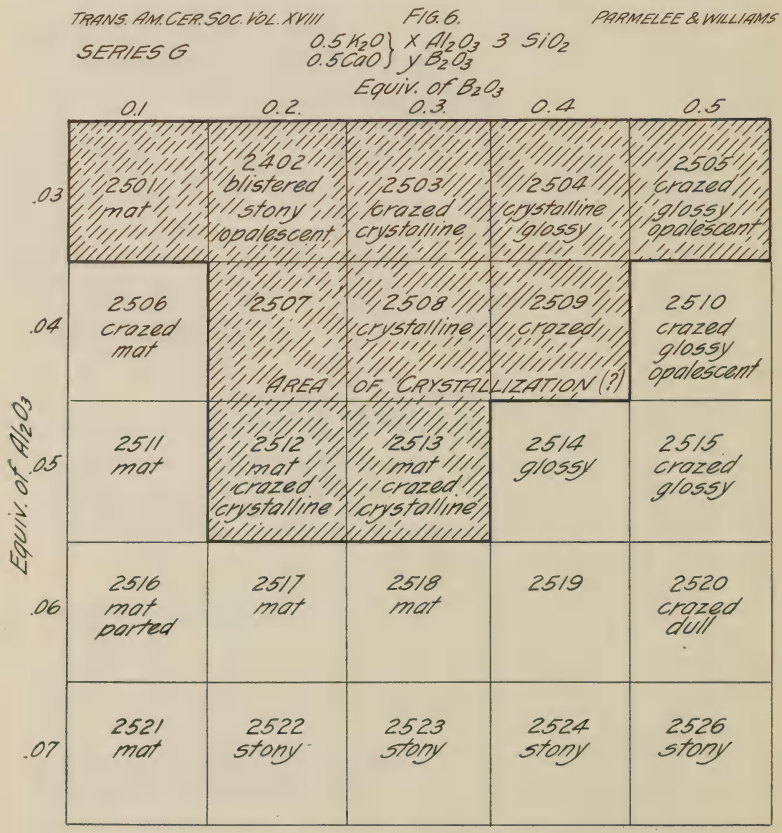
Color. Whiter glazes are had with the increase of silica.

Texture. The best surface texture, *i. e.* smoothness, freedom from pin-holes, egg-shell, etc., is had with lowest silica at

any boric acid content. Matness develops with a decrease of boric acid at any silica content.

The best mat is found, naturally, with the lowest silica and lowest boric acid content.

Crystallization. The glazes found within the area indicated



on Fig. 5 show surface markings which are apparently of a crystalline character.

Best glazes. The best glazes are those containing four or five equivalents of silica and 0.2 to 0.5 equivalents of boric acid. Glazes Nos. 2421 and 2425 are very good, excepting for faint surface markings.

Gloss. Those glazes containing less than 0.3 equivalents of boric acid are dull. Figure 5.

Series G. Crazing. Many of the glazes are crazed.

Gloss. The glossy glazes are found among those having 0.4 and 0.5 equivalents boric acid. Nos. 2510 and 2515 are good glossy glazes.

Opalescence. The glossy glazes bordering on the area of crystallization show opalescence.

Texture. Mats are found among those glazes having 0.3 equivalents or less of boric acid. As the alumina content increases, the mat area increases. Glaze No. 2517 is a superior mat. Fig. 6.

Series H. Crazing occurs on only one, No. 2547.

Crystalline area. In glazes having 0.3, 0.4 and 0.5 equivalents of alumina, in passing from low to high boric acid there is an area which appears to be marked by indications of crystallization. As the alumina content increases, this area decreases. Its boundaries are indicated on the chart. Those glossy glazes which border this area contain whitish separations. Mats are found with all contents of alumina having 0.1 equivalents of boric acid. As the boric acid content increases, the area of matness decreases.

Best glazes are found in the area indicated on the chart, *i. e.*, among those having 0.5 and 0.6 equivalents of alumina and 0.3 to 0.5 equivalents of boric acid. Fig. 7.

Series I. Area of crystallization is well defined, although the crystalline character is not well developed.

Best glossy glaze is No. 2570. Glazes outside of crystalline area are very good, excepting No. 2551. Fig. 8.

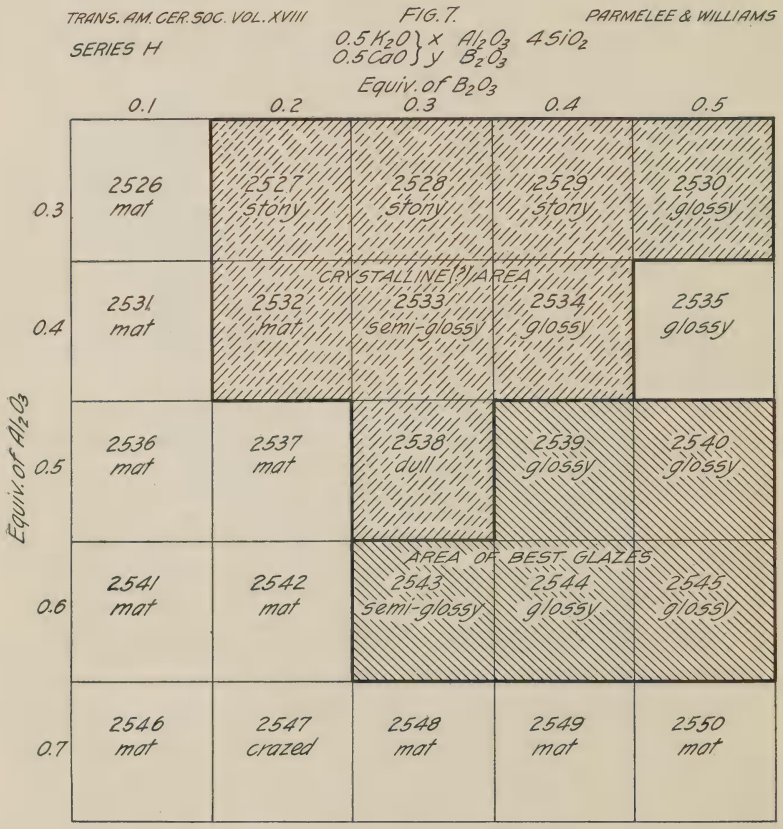
SUMMARY

R O Groups. We have found in these experiments that the best range of R O members was within the limits

0.4—0.6 K_2O
0.0—0.3 ZnO
0.4—0.6 CaO

Those glazes higher in calcium were dull, and those having more than 0.3 zinc oxide were likely to blister. High alkali content is usually accompanied by opalescence.

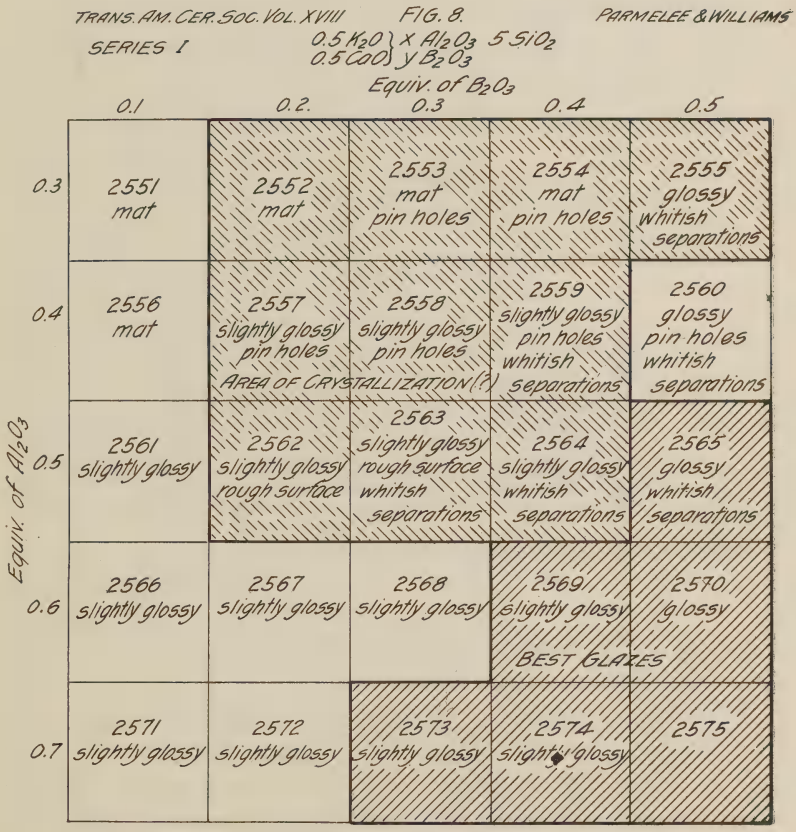
The use of barium introduced as 0.1 equivalent did not bring about any especially notable change in appearance or gloss.



The substitution of soda for half of the potash had the effect of extending somewhat the area of good glazes. The use of pearl ash in place of potassium nitrate did not bring about any marked change.

The R O compositian selected by us as the basis for experiments was chosen after inspection of trials fired in our

laboratory kiln. Subsequent burns in commercial kilns under longer period of firing showed that while the R O chosen was quite satisfactory, yet some other glazes containing zinc made even better appearance. The R O composition chosen, *i. e.*, glaze No. 2188, was selected solely upon the basis of the good appear-

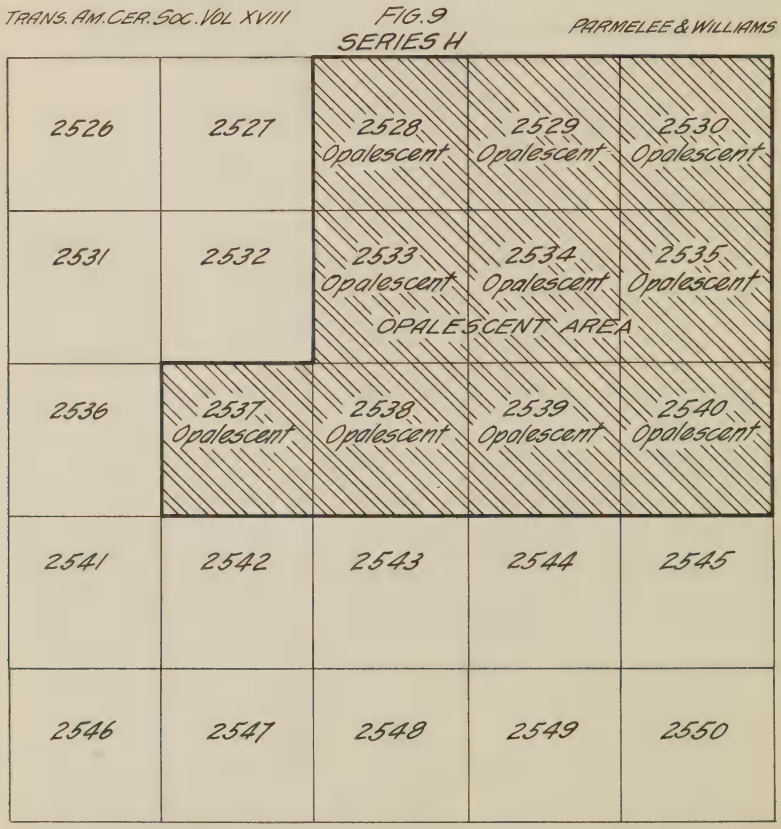


ance of the trials, and it is interesting to note that it falls within the limits assigned by Seger to glazes of this type suitable for lower temperatures, namely

$$\left.\begin{matrix} 0.6\ K_2O \\ 0.4\ CaO \end{matrix}\right\} \text{ and } \left\{\begin{matrix} 0.2\ K_2O \\ 0.8\ CaO \end{matrix}\right.$$

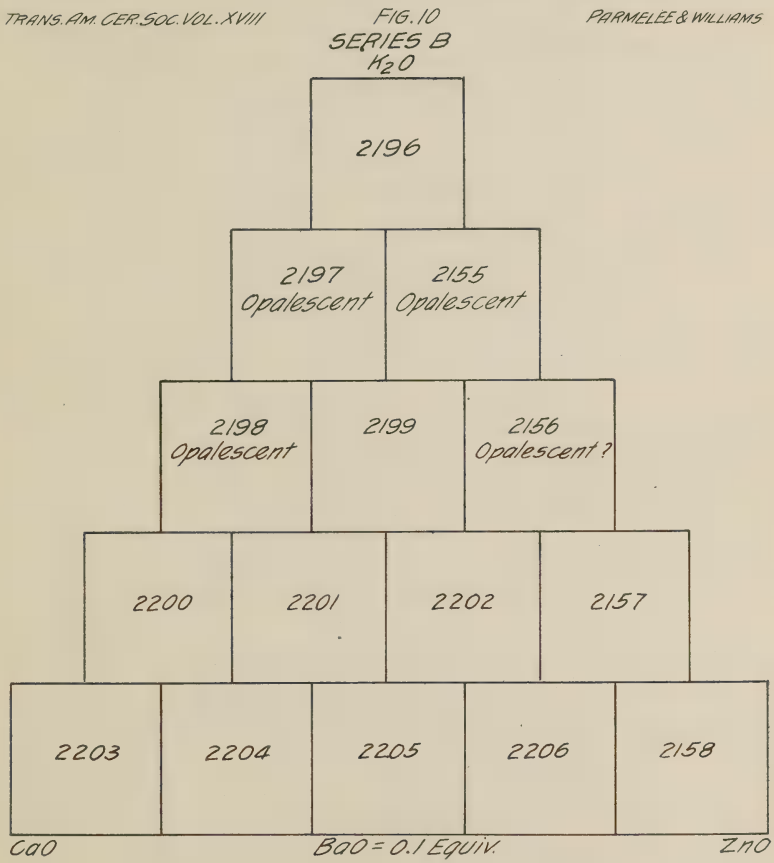
Silica. The silica content giving the best results as to gloss was five equivalents (See Series H).

Alumina. The amount of alumina to be recommended lies at either 0.5 or 0.6 equivalents for glossy glazes, although less than this may be used with 0.5 equivalents of boric oxide



present. With series having 3 and 4 equivalents of silica, 0.7 equivalents of alumina gives mat or immature glazes with all contents of boric oxide. Mat glazes are found most extensively in the series having the lowest silica content, and as the silica content increases, the number of glossy or semi-glossy glazes increases.

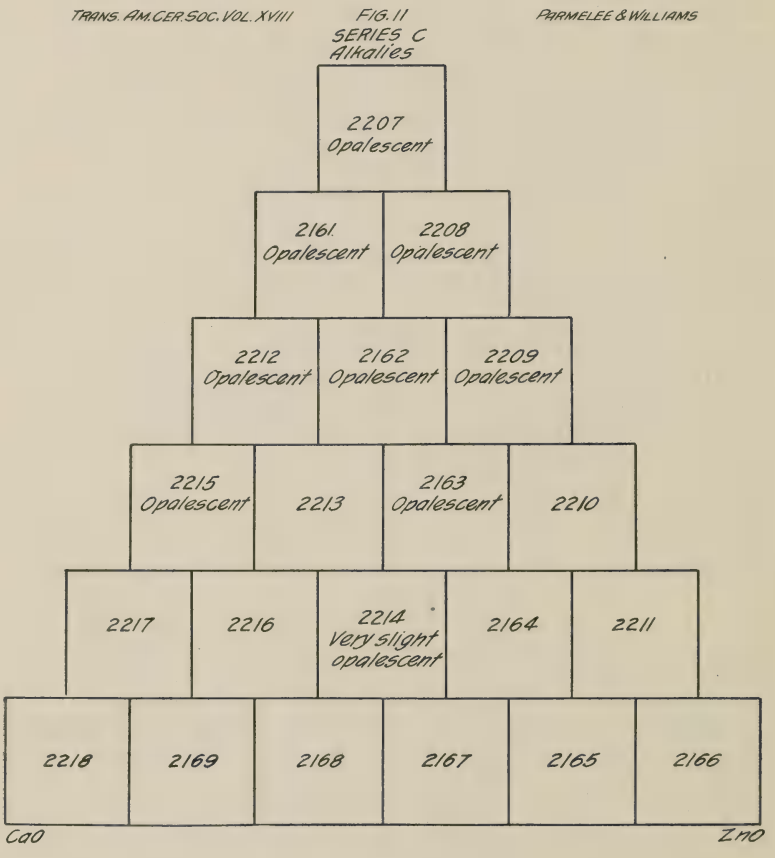
Boric Oxide. The effect of the increase of boric oxide in increasing the glossiness and fusibility of any glaze is apparent. The difference is not always as marked as one naturally expects. The most favorable B_2O_3 content is 0.5 equivalents.



Opalescence. As stated elsewhere, we have used this term to describe the appearance of samples which may not show a play of colors, but only a milky turbidity of the glaze. We have chosen to do this because these experiments appear to indicate that they are due to the same causes. We have also

noticed that opalescence may be caused by the occurrence of small blisters below the glaze surface.

With opalescence we have also found associated the dimming of the surface of the glaze in such a fashion as to have the appearance of a crystalline structure. Areas showing these appearances



are indicated as "crystalline" on the charts for Series F, G, H and I.

The most marked milky and opalescent appearance of the glaze we have obtained under the following conditions: among the high alkali glazes of Series A; among the glazes high in boric

acid in Series E; among the glazes high in boric acid, low in silica and low in alumina.

Additional experiments on opalescence were conducted in the same manner as used by Stull, (*Trans. Amer. Cer. Soc.* Vol. XII, 1910, page 129) that is, by preparing a biscuit tile with cup like depressions and filling the same with dry glaze, then firing in the kiln at cone 8. We can report results of only three series.

The experiment in the case of Series B developes a few instances of opalescence in a series which did not show any in the glaze trials. (See Figure 10).

Series C (Figure 11) shows an opalescence which does not appear on the glazed trials. The position of the opalescent area is as one would expect from study of similar series, that is, covering the portion of the field highest in alkalies.

The opalescence area in Series H (Figure 9) closely coincides with that observed on the glaze trials. (See previous chart of Series H).

Textures. Many of the glazes show an "egg shell" texture. Those that we have indicated as best are of course most free from such a fault. Those glazes which present the most satisfactory appearance are sometimes of doubtful value because milky or opalescent. It may be said that few of these glazes are as satisfactory in texture (and gloss) as the glazes containing lead now in use.

Crazing. Although these glazes were applied on bodies of different composition, yet, crazing was an unusual fault. It occured chiefly on glazes highest in alkalies, and the series (G) having the lowest silica content. In this series, apparently crazing decreases with an increase in alumina.

BATCH COMPOSITIONS

Frit 2160

$$\left.\begin{matrix} 0.25\ K_2O \\ 0.25\ Na_2O \\ 0.50\ CaO \end{matrix}\right\} \begin{matrix} 2.5\ SiO_2 \\ 1.25\ B_2O_3 \end{matrix}$$

	Batch weight	*Fritted weight*
Niter	20.02	9.4
Soda ash	10.6	6.2
Whiting	20.0	11.2
Boric acid	62.0	35.0
Flint	60.0	60.0
	172.8	121.8

Frit 2194

$$\left.\begin{matrix} 0.3571\ K_2O \\ 0.3571\ Na_2O \\ 0.2846\ CaO \end{matrix}\right\} \begin{matrix} 1.428\ SiO_2 \\ 0.714\ B_2O_3 \end{matrix}$$

	Batch weight	*Fritted weight*
Borax	95.5	50.5
Pearl ash	34.5	23.5
Whiting	20.0	11.2
Flint	60.0	60.0
	210.0	145.2

Frit 2195

$$\left.\begin{matrix} 0.25\ K_2O \\ 0.25\ Na_2O \\ 0.50\ CaO \end{matrix}\right\} \begin{matrix} 2.5\ SiO_2 \\ 1.25\ B_2O_3 \end{matrix}$$

	Batch weight	*Fritted weight*
Pearl ash	13.8	9.4
Borax	38.2	20.2
Whiting	20.0	11.2
Boric acid	37.2	21.0
Flint	60.0	60.0
	169.2	121.8

Frit 2144

$$\left.\begin{matrix} 0.6\ K_2O \\ 0.4\ CaO \end{matrix}\right\} \begin{matrix} 4.20\ SiO_2 \\ 1.50\ B_2O_3 \end{matrix}$$

	Batch weight	*Fritted weight*
Niter	121.2	56.4
Whiting	40.0	22.4
Flaky boric acid	186.0	105.0
Flint	252.0	252.0
	599.2	435.8

Frit 2154

$$\left.\begin{matrix}.33\ K_2O \\ .50\ CaO \\ 167\ BaO\end{matrix}\right\} \begin{matrix}2.33\ SiO_2 \\ 0.83\ B_2O_3\end{matrix}$$

	Batch weight	*Fritted weight*
Niter	40.4	18.8
Whiting	30.0	16.8
Barium carbonate	19.7	15.3
Flaky boric acid	62.0	35.0
Flint	84.0	84.0
	246.1	169.9

Frit 2159

$$\left.\begin{matrix}0.357\ K_2O \\ 0.357\ Na_2O \\ 0.286\ CaO\end{matrix}\right\} \begin{matrix}1.428\ SiO_2 \\ 0.714\ B_2O_3\end{matrix}$$

	Batch weight	*Fritted weight*
Niter	50.5	23.5
Soda ash	26.5	15.5
Whiting	20.0	11.2
Boric acid	62.0	35.0
Flint	60.0	60.0
	219.0	145.2

Frit 2386

$$\left.\begin{matrix}0.6\ K_2O \\ 0.4\ CaO\end{matrix}\right\} \begin{matrix}4.2\ SiO_2 \\ 0.3\ B_2O_3\end{matrix}$$

	Batch weight	*Fritted weight*
Pearl ash	82.8	56.4
Whiting	40.0	22.4
Boric acid	37.2	21.0
Flint	252.0	252.0
	412.0	351.8

Frit 2438

$$\left.\begin{matrix} 0.5\ K_2O \\ 0.5\ CaO \end{matrix}\right\} \begin{matrix} 2.0\ SiO_2 \\ 0.5\ B_2O_3 \end{matrix}$$

	Batch weight	*Fritted weight*
Pearl ash	13.8	9.4
Whiting	10.0	5.6
Boric acid	12.4	7.0
Flint	24.0	24.0
	60.2	36.0

Frit 2499

$$\left.\begin{matrix} 0.667\ K_2O \\ 0.333\ CaO \end{matrix}\right\} \begin{matrix} 2.0\ SiO_2 \\ 0.833\ B_2O_3 \end{matrix}$$

Pearl ash	55.2	37.6
Whiting	20.0	11.2
Boric acid	62.0	35.0
Flint	72.0	72.0
	209.2	155.8

Frit 2500

$$\left.\begin{matrix} 0.667\ K_2O \\ 0.333\ CaO \end{matrix}\right\} \begin{matrix} 2.0\ SiO_2 \\ 0.167\ B_2O_3 \end{matrix}$$

	Batch weight	*Fritted weight*
Pearl ash	55.2	37.6
Whiting	20.0	11.2
Boric acid	12.4	7.0
Flint	72.0	72.0
	159.6	127.8

Series A:

GLAZE NUMBERS	2145	2146	2147	2148	2149	2150	2151	2152	2153	2184
Frit No. 2144	145.0	145.0	145.0	145.0	145.0	145.0	145.0	145.0	145.0	145.0
Whiting	26.6	26.6	26.6	26.6	26.6	16.6	36.6	46.6	56.6	16.6
Zinc oxide	.....	8.1	16.2	24.3	32.4	40.5	24.3	16.2	8.1	8.1
Feldspar	222.8	167.1	111.4	55.7	.....	.....	.....	.....	.....	222.8
Florida clay	25.8	51.6	77.4	103.2	129.0	129.0	129.0	129.0	129.0	25.8
Flint	.....	24.0	48.0	72.0	96.0	96.0	96.0	96.0	96.0	.....

GLAZE NUMBERS	2185	2186	2187	2188	2189	2190	2191	2192	2193	2194
Frit No. 2144	145.0	145.0	145.0	145.0	145.0	145.0	145.0	145.0	145.0	145.0
Whiting	16.6	16.6	16.6	36.6	36.6	36.6	46.6	46.6	56.6	66.6
Zinc oxide	16.2	24.3	32.4	.....	8.1	16.2	.....	8.1	.....	.....
Feldspar	167.1	111.4	55.7	167.1	111.4	55.7	111.4	55.7	55.7	.....
Florida clay	51.6	77.4	103.2	51.6	77.4	103.2	77.4	103.2	103.2	129.0
Flint	24.0	48.0	72.0	24.0	48.0	72.0	48.0	72.0	72.0	96.0

Series B:

GLAZE NUMBERS	2155	2156	2157	2158	2196	2197	2198	2199	2200	2201
Frit No. 2154	169.9	169.9	169.9	169.9	169.9	169.9	169.9	169.9	169.9	169.9
Whiting	.....	.....	.....	.....	.....	10.0	20.0	10.0	30.0	20.0
Zinc oxide	8.1	16.2	24.3	32.4	.....	.....	.....	8.1	.....	8.1
Feldspar	167.1	111.4	55.7	.....	.....	167.1	111.4	111.4	55.7	55.7
Florida clay	51.6	76.8	103.2	129.0	222.8	51.6	77.4	77.4	103.2	103.2
Flint	24.0	48.0	72.0	96.0	25.8	24.0	48.0	48.0	72.0	72.0

GLAZE NUMBERS	2202	2203	2204	2205	2206
Frit No. 2154	169.9	169.9	169.9	169.9	169.9
Whiting	10.0	40.0	30.0	20.0	10.0
Zinc oxide	16.2	.....	8.1	16.2	24.3
Feldspar	55.7	.....	.....	.....	.....
Florida clay	103.2	129.0	129.0	129.0	129.0
Flint	72.0	96.0	96.0	96.0	96.0

Series C:

GLAZE NUMBERS	2161	2162	2163	2164	2165	2166	2167	2168	2169	2207
Frit No. 2159	145.2	145.2	96.8	48.4	.....	.....	.....	.....	.....	.....
Frit No. 2160	.....	.....	40.6	81.2	121.8	121.8	121.8	121.8	121.8	145.2
Goldings feldspar	27.9	.....	.....	.....	.....	.....	.....	.....	.....	55.7
Sparvetta feldspar	26.2	.....	.....	.....	.....	.....	.....	.....	.....	52.4
Zinc oxide	.....	8.1	16.2	24.3	32.4	40.5	24.3	16.2	8.1	.....
Whiting	20.0	20.0	20.0	20.0	20.0	10.0	30.0	40.0	50.0	10.0
Florida clay	103.2	129.0	129.0	129.0	129.0	129.0	129.0	129.0	129.0	77.4
Flint	96.0	120.0	120.0	120.0	120.0	120.0	120.0	120.0	120.0	72.0

GLAZE NUMBERS	2208	2209	2210	2211	2212	2213	2214	2215	2216	2217	2218
Frit No. 2159	145.2	145.2	96.8	48.4	145.2	96.8	48.4	96.8	48.4	48.4	.....
Frit No. 2160	.....	.....	40.6	81.2	.....	40.6	81.2	40.6	81.2	81.2	121.8
Goldings feldspar	27.9	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....
Sparvetta feldspar	26.2	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....
Zinc oxide	8.1	16.2	24.3	32.4	.....	8.1	16.2	.....	8.1	.....	.....
Whiting	10.0	10.0	10.0	10.0	30.0	30.0	30.0	40.0	40.0	50.0	60.0
Florida clay	103.2	129.0	129.0	129.0	129.0	129.0	129.0	129.0	129.0	129.0	129.0
Flint	96.0	120.0	120.0	120.0	120.0	120.0	120.0	120.0	120.0	120.0	120.0

Series D:

GLAZE NUMBERS	2219	2220	2221	2222	2223	2224	2225	2226	2227	2228	2229
Frit No. 2194	145.2	145.2	145.2	145.2	145.2	145.2	96.8	96.8	96.8	96.8	48.4
Frit No. 2195	.....	.....	.....	.....	.....	.....	40.6	40.6	40.6	40.6	81.2
Goldings feldspar	55.7	27.9	27.9	.....	.....	.....	.....	.....	.....	.....	.....
Sparvetta feldspar	52.4	26.2	26.2	.....	.....	.....	.....	.....	.....	.....	.....
Zinc oxide	.....	.....	8.1	.....	8.1	16.2	.....	8.1	16.2	24.3	.....
Whiting	10.0	70.0	10.0	30.0	20.0	10.0	40.0	30.0	20.0	10.0	50.0
Florida clay	77.4	103.2	103.2	129.0	129.0	129.0	129.0	129.0	129.0	129.0	129.0
Flint	72.0	96.0	96.0	120.0	120.0	120.0	120.0	120.0	120.0	120.0	120.0

GLAZE NUMBERS	2230	2231	2232	2233	2234	2235	2236	2237	2238	2239
Frit No. 2194	48.4	48.4	48.4	48.4	.....	.....	.....	.....	.....	.....
Frit No. 2195	81.2	81.2	81.2	81.2	121.8	121.8	121.8	121.8	121.8	121.8
Zinc oxide	8.1	16.2	24.3	32.4	.....	8.1	16.2	24.3	32.4	40.5
Whiting	40.0	30.0	20.0	10.0	60.0	50.0	40.0	30.0	20.0	10.0
Florida clay	129.0	129.0	129.0	129.0	129.0	129.0	129.0	129.0	129.0	129.0
Flint	120.0	120.0	120.0	120.0	120.0	120.0	120.0	120.0	120.0	120.0

Series E:

GLAZE NUMBERS	2387	2388	2389	2390	2391	2392	2393	2394	2395	2396	2397	2398
Frit No. 2144	108.7	72.5	36.2	.....	108.7	72.5	36.2	.....	108.7	72.5	36.2	.....
Frit No. 2386	29.3	58.6	87.9	117.3	29.3	58.6	87.9	117.3	29.3	58.6	87.9	117.3
Goldings feldspar	222.8	222.8	222.8	222.8	111.4	111.4	111.4	111.4	111.4	111.4	111.4	111.4
Zinc oxide	.....	.....	.....	.....	16.2	16.2	16.2	16.2	.....	.....	.....	.....
Whiting	26.6	26.6	26.6	26.6	26.6	26.6	26.6	26.6	46.6	46.6	46.6	46.6
Florida clay	25.8	25.8	25.8	25.8	77.4	77.4	77.4	77.4	77.4	77.4	77.4	77.4
Flint	.....	.....	.....	.....	48.0	48.0	48.0	48.0	48.0	48.0	48.0	48.0

GLAZE NUMBERS	2402	2403	2404	2405	2406	2407	2408	2409	2410	2411	2412	2413
Frit No. 2144	108.7	72.5	36.2	.....	108.7	72.5	36.2	.....	108.7	72.5	36.2	.....
Frit No. 2386	29.3	58.6	87.9	117.3	29.3	58.6	87.9	117.3	29.3	58.6	87.9	117.3
Goldings feldspar	167.1	167.1	167.1	167.1	167.1	167.1	167.1	167.1	111.4	111.4	111.4	111.4
Zinc oxide	.....	.....	.....	.....	8.1	8.1	8.1	8.1	8.1	8.1	8.1	8.1
Whiting	36.6	36.6	36.6	36.6	36.6	.....	.....	.....	36.6	36.6	36.6	36.6
Florida clay	51.6	51.6	51.6	51.6	51.6	51.6	51.6	51.6	77.4	77.4	77.4	77.4
Flint	24.0	24.0	24.0	24.0	24.0	24.0	24.0	24.0	48.0	48.0	48.0	48.0

Series F:

GLAZE NUMBERS	2418	2419	2420	2421	2422	2423	2424	2425	2426	2427
Frit No. 2144	144.9	144.9	144.9	144.9	108.7	108.7	108.7	108.7	72.5	72.5
Frit No. 2386	.....	.....	.....	.....	29.3	29.3	29.3	29.3	58.6	58.6
Whiting	36.6	36.6	36.6	36.6	36.6	36.6	36.6	36.6	36.6	36.6
Feldspar	167.1	167.1	167.1	167.1	167.1	167.1	167.1	167.1	167.1	167.1
Florida clay	51.6	51.6	51.6	51.6	51.6	51.6	51.6	51.6	51.6	51.6
Flint	204.0	144.0	84.0	24.0	204.0	144.0	84.0	24.0	204.0	144.0

GLAZE NUMBERS	2428	2429	2430	2431	2432	2433	2434	2435	2436	2437
Frit No. 2144	72.5	72.5	36.2	36.2	36.2	36.2	.....	.....	.....	.....
Frit No. 2386	58.6	58.6	87.9	87.9	87.9	87.9	117.3	117.3	117.3	117.3
Whiting	36.6	36.6	36.6	36.6	36.6	36.6	36.6	36.6	36.6	36.6
Feldspar	167.1	167.1	167.1	167.1	167.1	167.1	167.1	167.1	167.1	167.1
Florida clay	51.6	51.6	51.6	51.6	51.6	51.6	51.6	51.6	51.6	51.6
Flint	84.0	24.0	204.0	144.0	84.0	24.0	204.0	144.0	84.0	24.0

Series G:

GLAZE NUMBERS	2501	2502	2503	2504	2505	2506	2507	2508	2509	2510
Frit. No. 2499	.....	38.9	77.9	116.8	155.9	.....	38.9	77.9	116.8	155.8
Frit No. 2500	127.8	96.0	64.0	32.0	.....	127.8	96.0	64.0	32.0	.....
Whiting	30.0	30.0	30.0	30.0	30.0	30.0	30.0	30.0	30.0	30.0
Feldspar	55.7	55.7	55.7	55.7	55.7	55.7	55.7	55.7	55.7	55.7
Florida clay	51.6	51.6	51.6	51.6	51.6	77.4	77.4	77.4	77.4	77.4
Flint	48.0	48.0	48.0	48.0	48.0	36.0	36.0	36.0	36.0	36.0

GLAZE NUMBERS	2511	2512	2513	2514	2515	2516	2517	2518	2519	2520
Frit No. 2499	.....	38.9	77.9	116.8	155.9	.....	38.9	77.9	116.8	155.8
Frit No. 2500	127.8	96.0	64.0	32.0	.....	127.8	96.0	64.0	32.0	.....
Whiting	30.0	30.0	30.0	30.0	30.0	30.0	30.0	30.0	30.0	30.0
Feldspar	55.7	55.7	55.7	55.7	55.7	55.7	55.7	55.7	55.7	55.7
Florida clay	103.2	103.2	103.2	103.2	103.2	129.0	129.0	129.0	129.0	129.0
Flint	24.0	24.0	24.0	24.0	24.0	12.0	12.0	12.0	12.0	12.0

GLAZE NUMBERS	2521	2522	2523	2524	2525
Frit No. 2499	.....	38.9	77.9	116.8	155.8
Frit No. 2500	127.8	96.0	64.0	32.0	.....
Whiting	30.0	30.0	30.0	30.0	30.0
Feldspar	55.7	55.7	55.7	55.7	55.7
Florida clay	154.8	154.8	154.8	154.8	154.8
Flint	.....	.....	.....	.....	.....

Series H:

GLAZE NUMBERS	2526	2527	2528	2529	2530	2531	2532	2533	2534	2535
Frit No. 2499	.....	38.9	77.9	116.8	155.8	.....	38.9	77.9	116.8	155.8
Frit No. 2500	127.8	96.0	64.0	32.0	.....	127.8	96.0	64.0	32.0	.....
Whiting	30.0	30.0	30.0	30.0	30.0	30.0	30.0	30.0	30.0	30.0
Feldspar	55.7	55.7	55.7	55.7	55.7	55.7	55.7	55.7	55.7	55.7
Florida clay	51.6	51.6	51.6	51.6	51.6	77.4	77.4	77.4	77.4	77.4
Flint	108.0	108.0	108.0	108.0	108.0	96.0	96.0	96.0	96.0	96.0

GLAZE NUMBERS	2536	2537	2538	2539	2540	2541	2542	2543	2544	2545
Frit No. 2490	.....	38.9	77.9	116.8	155.8	.....	38.9	77.9	116.8	155.8
Frit No. 2500	127.8	96.0	64.0	32.0	.....	127.8	96.0	64.0	32.0	.....
Whiting	30.0	30.0	30.0	30.0	30.0	30.0	30.0	30.0	30.0	30.0
Feldspar	55.7	55.7	55.7	55.7	55.7	55.7	55.7	55.7	55.7	55.7
Florida clay	103.2	103.2	103.2	103.2	103.2	129.0	129.0	129.0	129.0	129.0
Flint	84.0	84.0	84.0	84.0	84.0	72.0	72.0	72.0	72.0	72.0

GLAZE NUMBERS	2546	2547	2548	2549	2550
Frit No. 2499	.....	38.9	77.9	116.8	155.8
Frit No. 2500	127.8	96.0	64.0	32.0	.....
Whiting	30.0	30.0	30.0	30.0	30.0
Feldspar	55.7	55.7	55.7	55.7	55.7
Florida clay	154.8	154.8	154.8	154.8	154.8
Flint	60.0	60.0	60.0	60.0	60.0

Series I:

GLAZE NUMBERS	2551	2552	2553	2554	2555	2556	2557	2558	2559	2560
Frit No. 2499	.....	38.9	77.9	116.8	155.8	.....	38.9	77.9	116.8	155.8
Frit No. 2500	127.8	96.0	64.0	32.0	.....	127.8	96.0	64.0	32.0	.....
Whiting	30.0	30.0	30.0	30.0	30.0	30.0	30.0	30.0	30.0	30.0
Feldspar	55.7	55.7	55.7	55.7	55.7	55.7	55.7	55.7	55.7	55.7
Florida clay	51.6	51.6	51.6	51.6	51.6	77.4	77.4	77.4	77.4	77.4
Flint	168.0	168.0	168.0	168.0	168.0	156.0	156.0	156.0	156.0	156.0

GLAZE NUMBERS	2561	2562	2563	2564	2565	2566	2567	2568	2569	2570
Frit No. 2499	.....	38.9	77.9	116.8	155.8	.....	38.9	77.9	116.8	155.8
Frit No. 2500	127.8	96.0	64.0	32.0	.....	127.8	96.0	64.0	32.0	.....
Whiting	30.0	30.0	30.0	30.0	30.0	30.0	30.0	30.0	30.0	30.0
Feldspar	55.7	55.7	55.7	55.7	55.7	55.7	55.7	55.7	55.7	55.7
Florida clay	103.2	103.2	103.2	103.2	103.2	129.0	129.0	129.0	129.0	129.0
Flint	144.0	144.0	144.0	144.0	144.0	132.0	132.0	132.0	132.0	132.0

GLAZE NUMBERS	2571	2572	2573	2574	2575
Frit No. 2499	.....	38.9	77.9	116.8	155.8
Frit No. 2500	127.8	96.0	64.0	32.0	.....
Whiting	30.0	30.0	30.0	30.0	30.0
Feldspar	55.7	55.7	55.7	55.7	55.7
Florida clay	154.8	154.8	154.8	154.8	154.8
Flint	120.0	120.0	120.0	120.0	120.0

A STUDY OF KANSAS SHALES

BY PAUL TEETOR

The ten shales selected for this study occur in the Pennsylvanian formation and were selected from deposits which are being used at the present time for the manufacture of various grades of clay products.

The primary object of this investigation was to determine the viscosity of these typical Kansas shales at seven different kiln temperatures. The temperatures selected for this work were Seger cones 06, 03, 1, 3, 5, 7 and 8. The kiln was heated up at a fairly rapid irregular rate until 200° below the finishing temperature, then the heating was increased at a regular rate of 20° per hour until the final temperature was reached, and this temperature held for one hour. This temperature regulation was easily executed by the use of natural gas as fuel and a platinum pyrometer for recording the temperature.

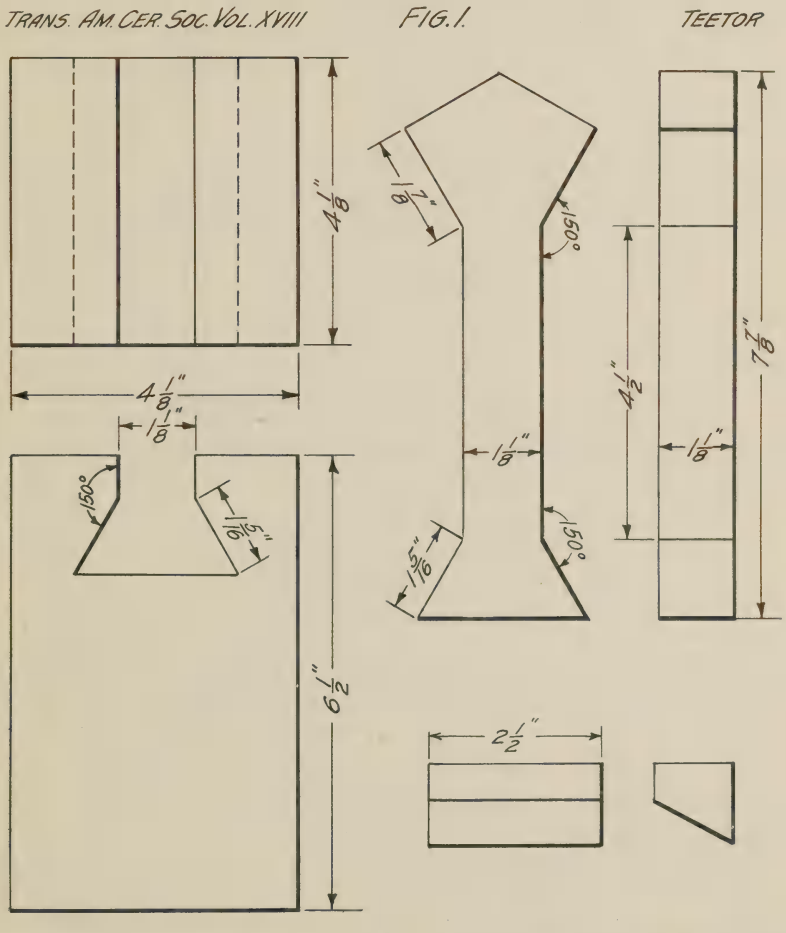
In addition to viscosity determination the following data was determined.

Drying shrinkage.
Burning shrinkage-temperature relation.
Porosity-temperature relation.
Microscopic examination of the burned specimens.

Shale No. 230 is a hard blue shale mined at a depth of 880 feet below the surface of the ground. It requires quite a bit of time to develop sufficient plasticity to enable it to be molded into small bricketts. This shale is used for the manufacture of common stiff-mud building brick.

Shale No. 231 is a hard blue shale similar to No. 230 as regards its plasticity. This shale is used for manufacturing hollow building tile. The curves shown in this paper indicate that this shale could also be used for the manufacture of vitrified brick.

Shale No. 232 is a blue shale underlying No. 233, which is a yellow shale, while No. 234 is a mixture of Nos. 232 and 233, and is being used for the manufacture of paving brick. These



shales develop very good plasticity upon the addition of the proper amount of water.

Shale No. 235 is a hard blue shale requiring time to develop a small amount of plasticity. This shale is being used for the manufacture of vitrified paving brick.

Shale No. 236 is a blue shale with good plasticity and is used for manufacturing drain tile.

Shale No. 237 is a blue shale with good plasticity and has been used as a portion of the mixture in the manufacture of vitrified brick.

Shale No. 238 is a blue shale with excessive plasticity, which has been used for the manufacture of common building brick and drain tile.

Shale No. 239 is a yellow shale with good plasticity and has been used for the manufacture of vitrified paving brick.

The test pieces used for the viscosity determination are very similar to those used by the U. S. Bureau of Standards in their work on the viscosity of shales. The shape of piece as well as the weight attached is shown in the sketch in Figure 1. The fire clay weight suspended to the bottom of the test piece was attached to give a tensile load of five pounds per square inch of cross section of the test piece, after it had been biscuited to a temperature of 850°C. These test pieces with their attached weights were suspended in the kiln in such a manner that the kiln gases could circulate freely about them. The lower edge of the test piece was ten inches above the floor of the kiln. The bricketts used for the porosity determination, as well as shrinkage temperature relations and for specimens for microscopic examination, were 2 in. by 2 in. by 1 in. The data secured from these determinations have been tabulated in Tables I, II and III and are shown graphically in Figures 2, 3, and 4, and are self-explanatory.

It will be observed that the curves in Figure 3 appear to bear some relation to those in Figure 2, but it is also noticed that there is a more distinct difference between the curves of the shales suited for vitrified ware and those not suited for such ware. In Figure 3, the curves of the shales which are suited for the manufacture of vitrified brick do not show any abrupt break, or decrease in the viscosity, below a temperature of Seger cone 5. It should be remembered that shale No. 237 does not produce a good grade of vitrified ware when used by itself. The curves of the shales not suited for vitrified ware show a break in the viscosity curve, or a decrease in the viscosity, at

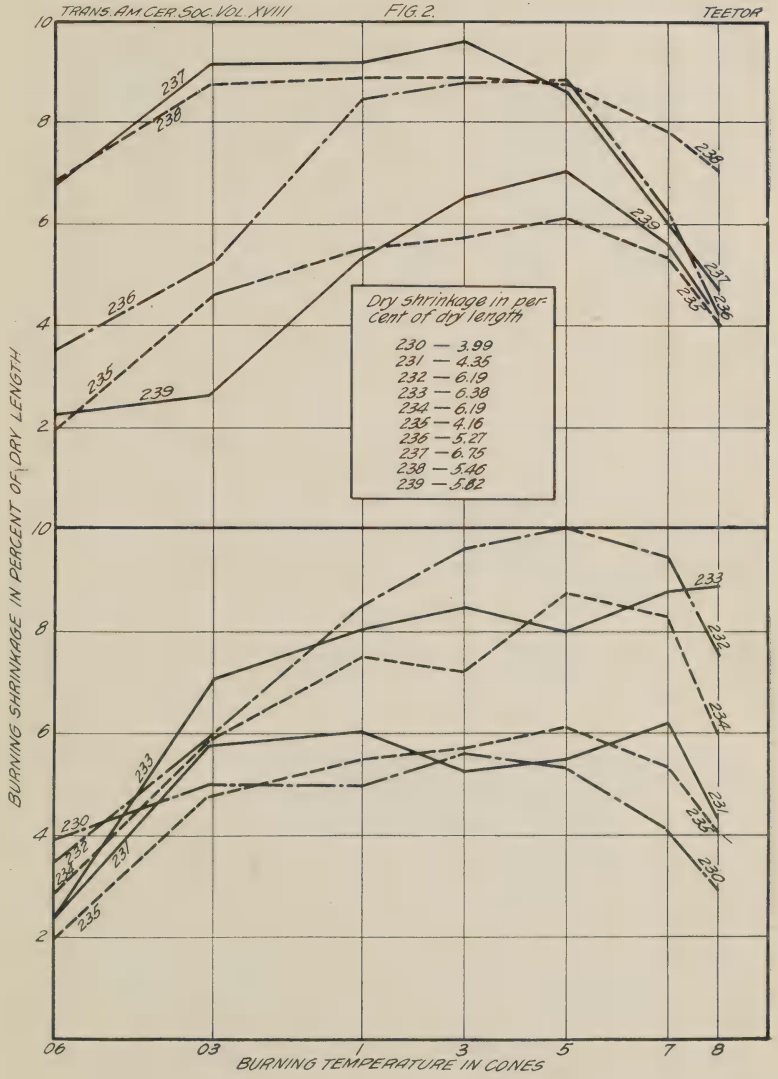

TRANS. AM CER. SOC. VOL. XVIII
FIG. 2.
TEETOR
BURNING SHRINKAGE IN PERCENT OF DRY LENGTH
BURNING TEMPERATURE IN CONES
06
03
1
3
5
7
8
2
4
6
8
10
Dry shrinkage in per cent of dry length
230 — 3.99
231 — 4.35
232 — 6.19
233 — 6.38
234 — 6.19
235 — 4.16
236 — 5.27
237 — 6.75
238 — 5.46
239 — 5.82

TABLE I. PERCENT BURNING-SHRINKAGE

SAMPLE NUMBER	BURNING TEMPERATURE IN CONES						
	06	03	1	3	5	7	8
230	3.90	5.00	5.01	5.62	5.38	4.15	2.96
231	3.66	5.78	6.05	5.29	5.41	6.20	4.36
232	3.47	5.92	8.51	9.66	10.04	9.48	7.58
232	3.53	7.14	8.07	8.50	8.00	8.84	8.91
234	2.83	5.93	7.50	7.22	8.77	8.31	5.91
235	2.00	4.67	5.53	5.74	6.15	5.35	4.06
236	3.50	3.50	8.46	8.85	8.83	6.24	4.10
237	6.83	9.15	9.18	9.62	8.64	6.05	4.72
238	6.85	8.78	8.89	8.88	8.82	7.85	7.10
239	2.28	2.64	5.48	6.51	7.07	5.69	4.05

TABLE II. PERCENT APPARENT POROSITY

SAMPLE NO.	BURNING TEMPERATURE IN CONES							
	850°C	06	03	1	3	5	7	8
230	31.30	19.58	8.23	5.75	5.06	5.34	5.38	5.41
231	29.64	19.71	13.97	9.46	6.30	5.94	3.57	2.20
232	34.89	26.85	15.48	10.87	7.53	.46	.13	.13
233	35.31	25.32	12.99	9.91	8.88	6.54	.44	.52
234	31.68	23.55	13.90	10.15	9.16	2.43	1.45	4.82
235	27.30	20.55	11.20	7.62	6.25	5.27	1.11	.81
236	32.45	23.14	18.88	7.71	3.79	.50	1.10	2.38
237	29.62	12.78	.31	.25	.32	1.31	3.60	8.25
238	32.51	13.32	4.01	2.49	2.77	2.40	2.26	2.42
239	28.56	21.84	19.15	10.15	7.71	5.93	1.19	3.10

TABLE III. PERCENT ELONGATION, BURNING TEMPERATURE IN CONES

SAMPLE NO.	BURNING TEMPERATURE IN CONES						
	06	03	1	3	5	7	8
230	—2.40	—3.50	—3.15	—1.95	— .60	+p	.....
231	—2.10	—2.95	—3.20	—3.25	—2.70	— .90	p
232	—1.70	—2.70	—3.60	—4.25	—4.50	—1.20p	.....
233	—2.10	—2.75	—2.70	—2.45	—2.45	— .90	+ .14p
234	—2.20	—2.75	—2.80	—2.85	—2.75	— .25	+2.45p
235	—2.00	—2.55	—3.00	—2.85	—2.40	p	.....
236	—2.20	—3.30	—3.70	—2.95	— .90	+ .47p	.....
237	—4.70	—5.95	—5.60	—5.05	—3.75	— .55	+ .80p
238	—5.30	—6.55	—6.35	—6.10	—5.65	—3.85	—2.00
239	—1.40	—2.00	—2.40	—2.80	—3.20	—2.55	+ .85p

p=pulled apart.

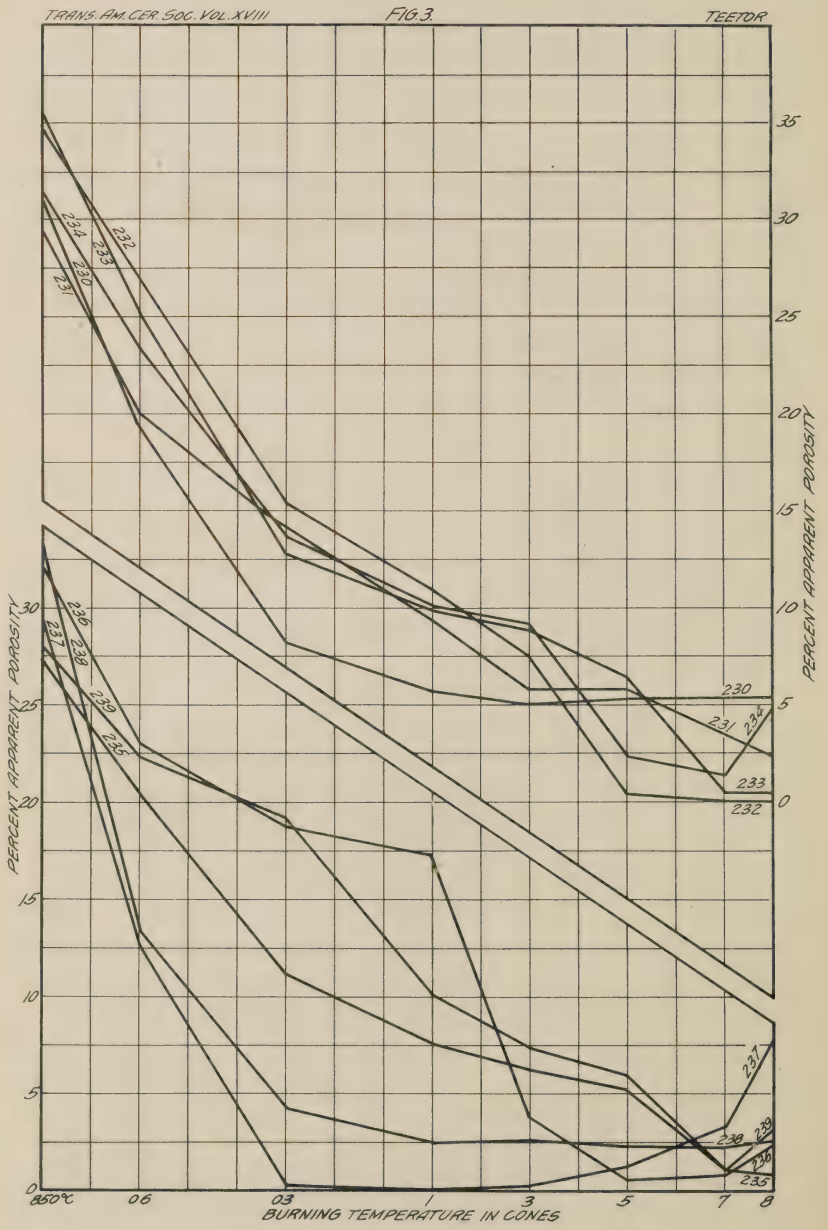
some temperature below Seger cone 5. This relation seems to work out very well with these particular shales, but the number of shales experimented with is entirely too small to justify drawing any general conclusions in this connection. It is to be noticed, however, that the critical point indicating the value of the shale is the point showing where the decided decrease in viscosity begins, rather than the point where the test piece pulls in two.

The microscopic examination of these shales consisted of examining thin sections prepared from these specimens which were burned at a temperature of Seger cone 06, 3, and 8, with the exception of shale No. 239 which was examined after it had been burned to all of the temperatures mentioned ealier in the paper.

These thin sections were examined with the regular petrographic microscope, and the micro-photographs reproduced in the paper show thin sections of the shales in polarized light with and without crossed nicols. The specimens represented were burned to Seger cone 3. This temperature was selected because, for the majority of the shales, it was the best maturing temperature.

The micro-photograph of shale No. 230 shows very clearly the outline of the various coarse particles of the shale as formed during the crushing process. Due to the slow development of plasticity, it has not been thoroughly cemented together, and a resulting large amount of pore space is easily seen. It may also be noticed in the photograph showing the section under crossed nicols, that there is an extremely small amount of siliceous matter or other crystalline material transmitting the light. When the same shale is burned at a higher temperature and examined microscopically, it may be seen that the large coarse particles of shale have become more cemented together and the original pore space is greatly decreased, but also, that there is a rather large amount of circular air holes present, due, undoubtedly, to production of a bleb structure.

Shale No. 231 bears a certain resemblance to No. 230 under the microscope, inasmuch as there is not a large amount of siliceous or other crystalline matter visible with crossed nicols.


TRANS. AM. CER. SOC. VOL. XVIII
FIG. 3.
TEETOR
PERCENT APPARENT POROSITY
PERCENT APPARENT POROSITY
BURNING TEMPERATURE IN CONES
850°C
06
03
1
3
5
7
8
0
5
10
15
20
25
30
35
230
231
232
233
234
235
236
237
238
239

This section, however, shows the material to be more compact and does not show the presence of any bleb structure when burned at a temperature of cone 8.

Shale No. 232 shows the usual amount of compactness of a good shale and also a large amount of very fine-grained quartz, and a relatively large amount of mica. The mica is easily visible at a temperature of cone 06, and cone 3, but is not recognizable at a temperature of cone 8. It will be noticed that there are practically no large pieces of quartz but a large amount of extremely fine quartz. The photographs represented here in each case show a magnification of 40 diameters.

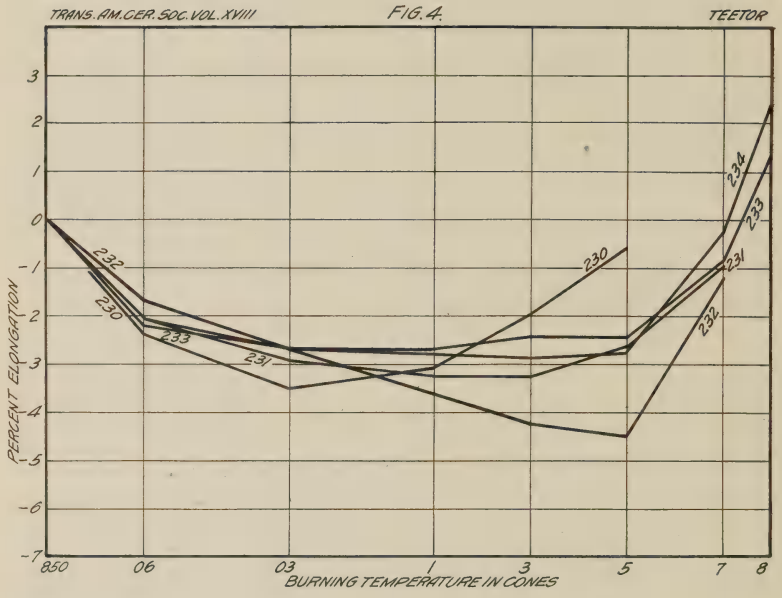
Shale No. 233 is very similar to No. 232 excepting that there is a smaller amount of mica and some larger quartz particles visible. A trace of the mica can still be seen at a temperature of cone 8. Also, at this temperature a few rounded holes are to be seen, but they are so few in number that it is doubtful whether they are due to the formation of any bleb structure.

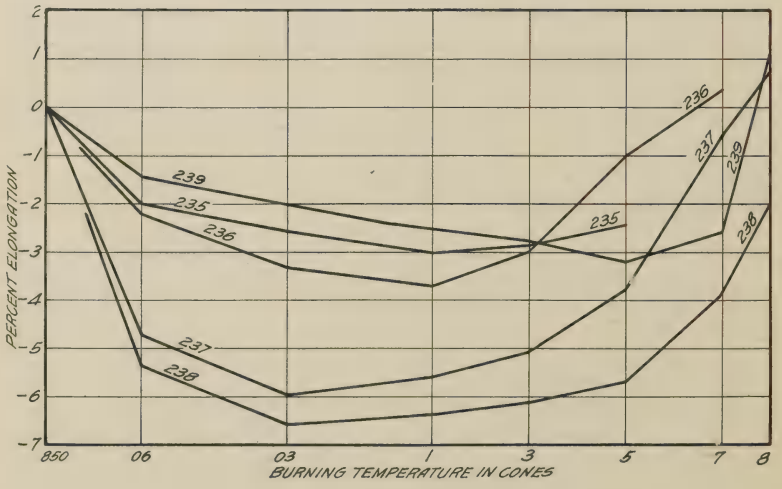
Shale No. 234 is a mixture of the two preceding shales and shows no additional or different qualities.

Shale No. 235 shows the usual amount of quartz both large and small sizes as well as some highly ferruginous areas which at the maturing temperature of the shale have contracted more than the main portion of the shale and have left considerable pore space around the ferruginous mass.

Shale No. 236 gives the same general appearance under the microscope as the preceding shale, insofar as can be seen, and presents no data yielding any light as to the reason for the poor qualities of this shale.

In shale No. 237 will be seen some very large quartz particles which show that at this temperature there has been no rounding due to heat, as there are a number of very sharp angles, almost needle points, still present on these quartz particles. These sharp angular particles show up more distinctly on the photograph with the crossed nicols, and when burned at a temperature of cone 8, there are small areas of yellowish glassy material present in which are imbedded a thin net work of very minute needles. I have not been able to determine accurately the composition of these needles, but some of their properties


TRANS. AM. CER. SOC. VOL. XVIII
FIG. 4.
TEETOR
PERCENT ELONGATION
3
2
1
0
-1
-2
-3
-4
-5
-6
-7
232
230
233
231
230
234
233
231
232
850
06
03
1
3
5
7
8
BURNING TEMPERATURE IN CONES


PERCENT ELONGATION
2
1
0
-1
-2
-3
-4
-5
-6
-7
239
235
236
237
238
236
237
239
238
235
850
06
03
1
3
5
7
8
BURNING TEMPERATURE IN CONES

indicate that they have a composition of sillimanite. At a temperature of cone 8, there is a large amount of pore space present due entirely to the production of the bleb structure.

Shale No. 238 is a very plastic ferruginous shale, and its appearance under the microscope is very similar to shale No. 230, except that the various coarse particles and the resulting pore space are not to be seen. The similarity consists in the fact that there is an extremely small amount of fine siliceous material present in the shale, which is shown more distinctly in the micro-photograph with the crossed nicols. In the photograph without the crossed nicols, is quite plainly shown a very small area where the highly ferruginous portion has shrunk away and left a very large pore space surrounding it. This shale at higher temperatures shows a small amount of bleb structure.

Shale No. 239 is at once seen to be a rather siliceous material showing a number of areas made up of a conglomerate of smaller quartz particles. The pore spaces in the shale are all of irregular outline, and there is no indication of a bleb structure. At a temperature of cone 8, there are some areas of a yellow glassy material in which are imbedded a net work of small needles of the same appearance as those present in shale No. 237.

Conclusion. Insofar as the ten shales studied are concerned, this investigation indicates that the viscosity temperature curve is capable of giving additional data to aid in differentiating between shales for the manufacture of vitrified products, such as paving brick, and those not suited for such products.

The microscopic examination indicates that the microscope is of more value as an aid in improving a product that is being manufactured, rather than to determine the value of a shale.

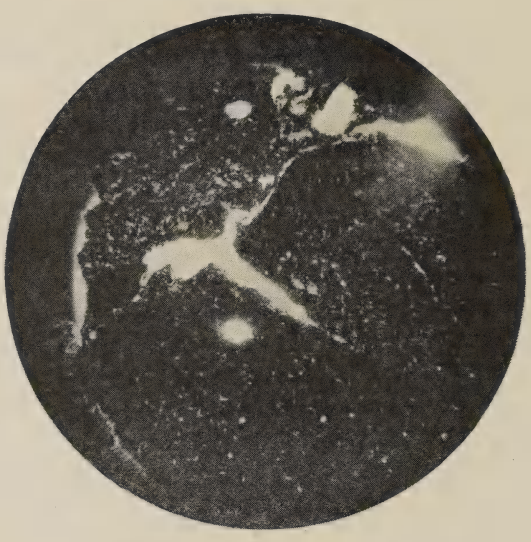


FIG. 5a — Clay No. 230, 40 diam.

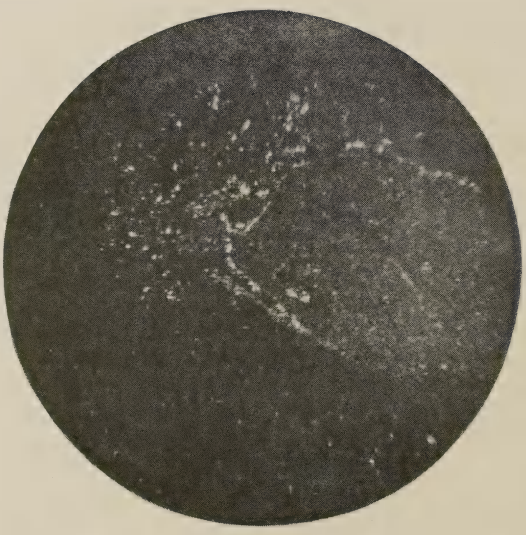


FIG. 5b — Clay No. 230, Crossed Nicols.

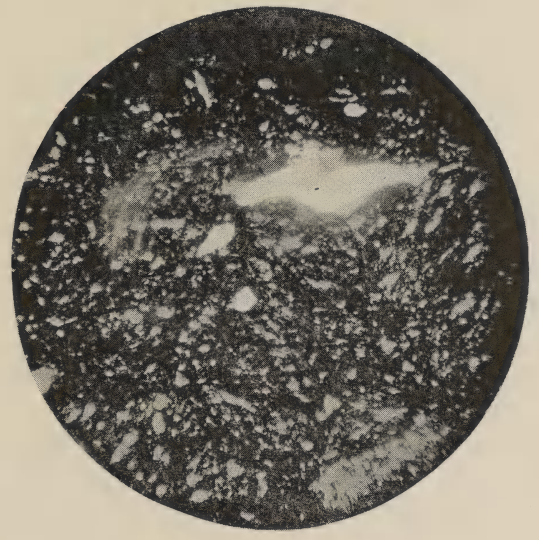


FIG. 6a — Clay No. 231.

FIG. 6b — Clay No. 231, Crossed Nicols.

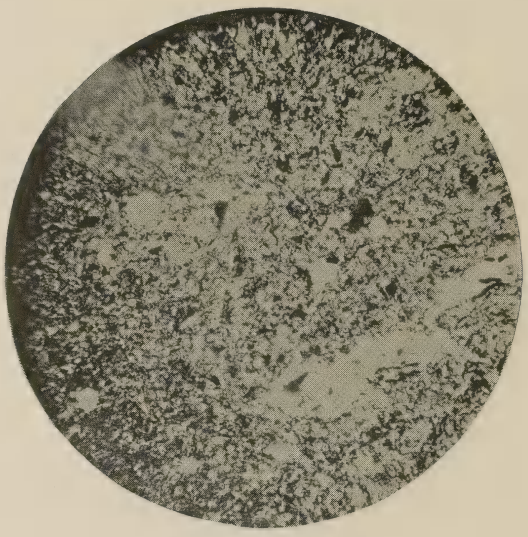


FIG. 7a — Clay No. 232.

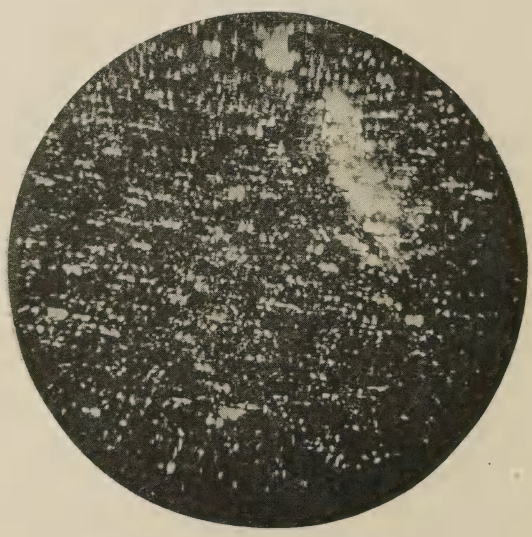


FIG. 7b — Clay No. 232, Crossed Nicols.

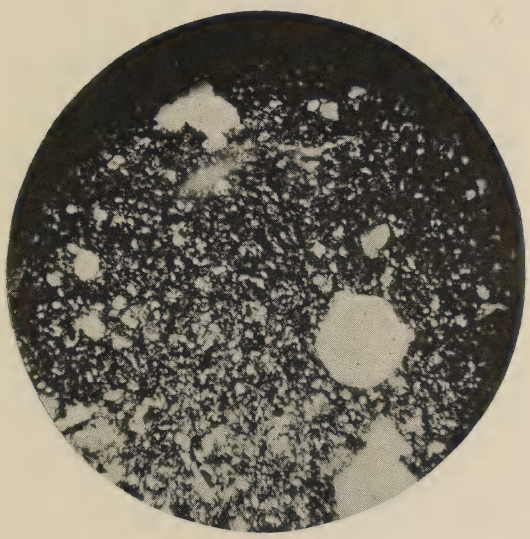


FIG. 8a — Clay No. 233.

FIG. 8b — Clay No. 233. Crossed Nicols.

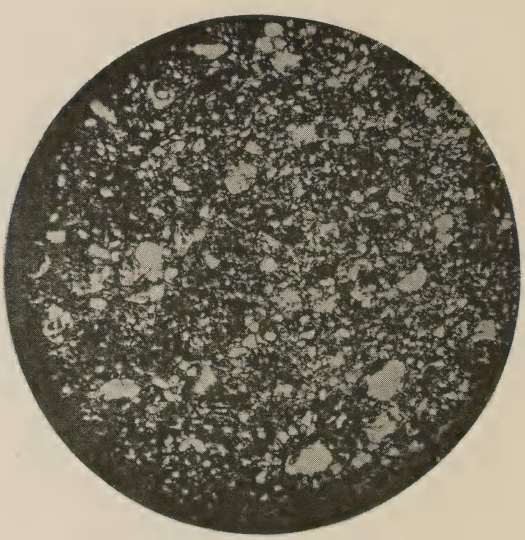


FIG. 9a — Clay No. 234.

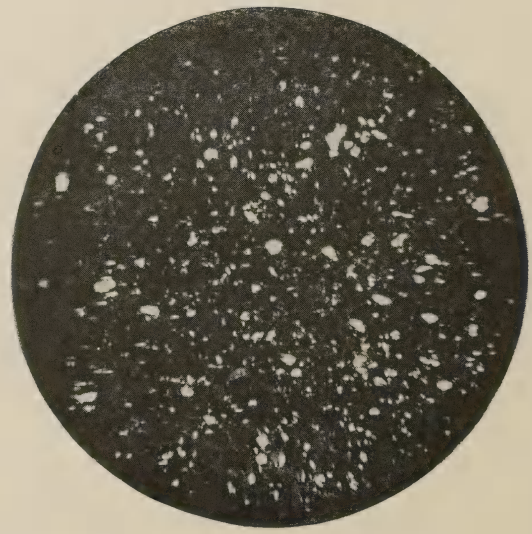


FIG. 9b — Clay No. 234, Crossed Nicols.

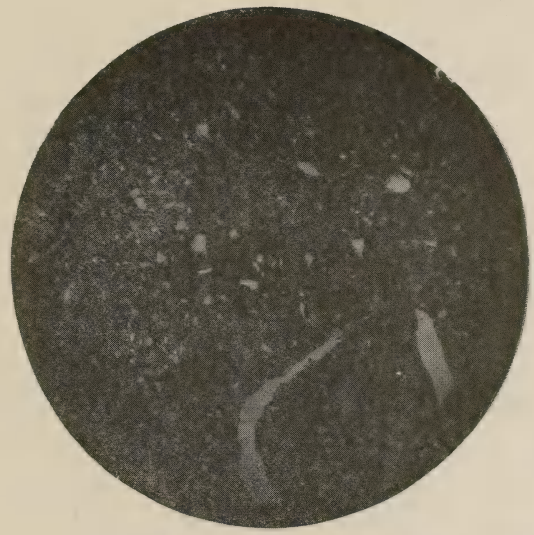


FIG. 10a — Clay No. 235.

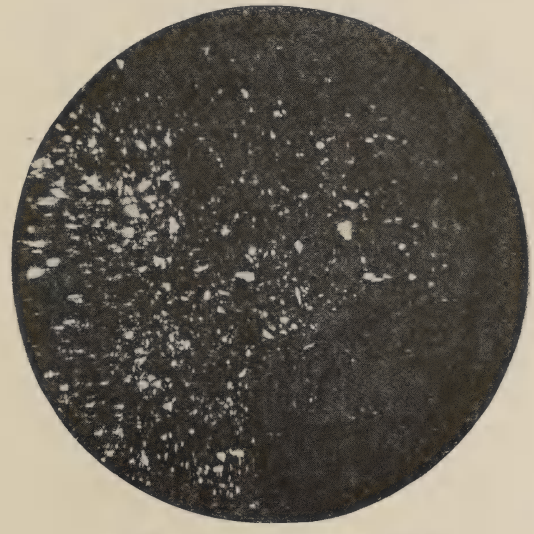


FIG. 10b — Clay No. 235, Crossed Nicols.

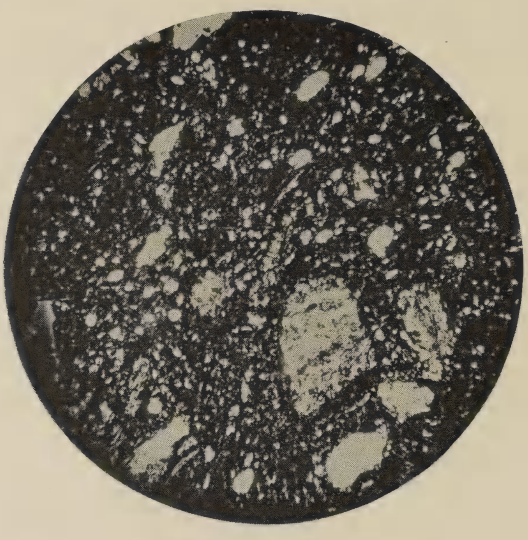


FIG. 11a — Clay No. 236.

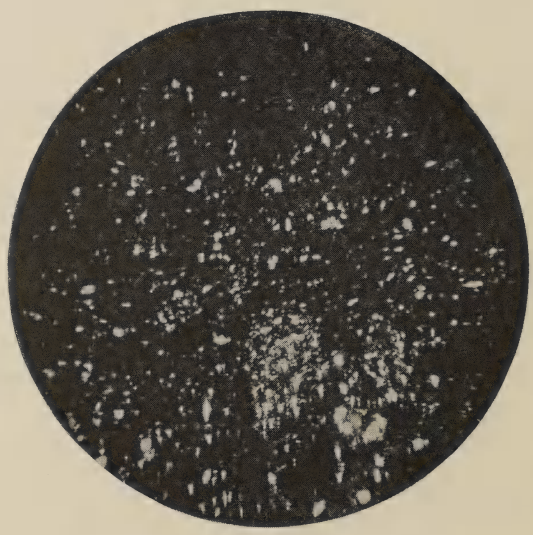


FIG. 11b — Clay No. 236, Crossed Nicols.

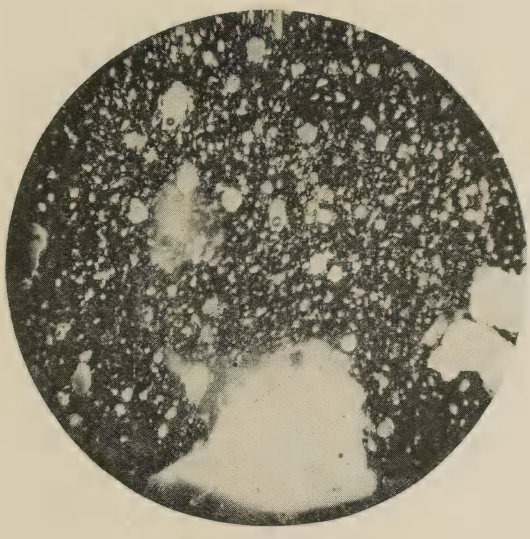


FIG. 12a — Clay No. 237.

FIG. 12b — Clay No. 237, Crossed Nicols.

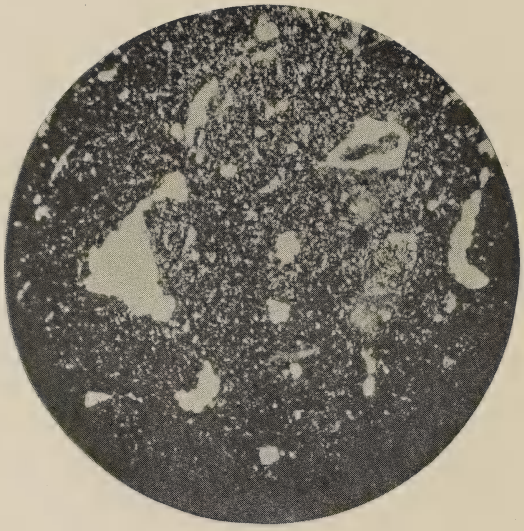


FIG. 13a — Clay No. 238.

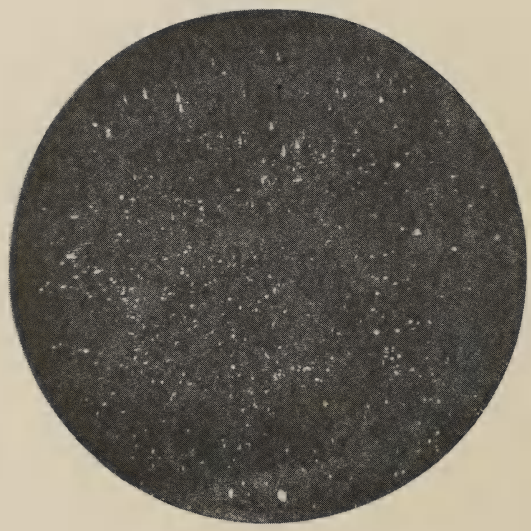


FIG. 13b — Clay No. 238, Crossed Nicols.

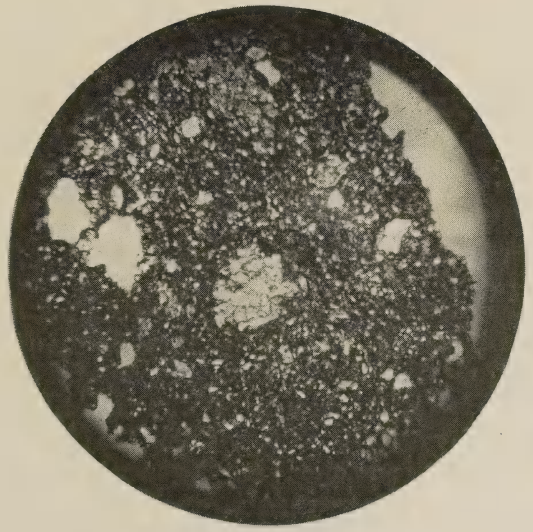


FIG. 14a — Clay No. 239.

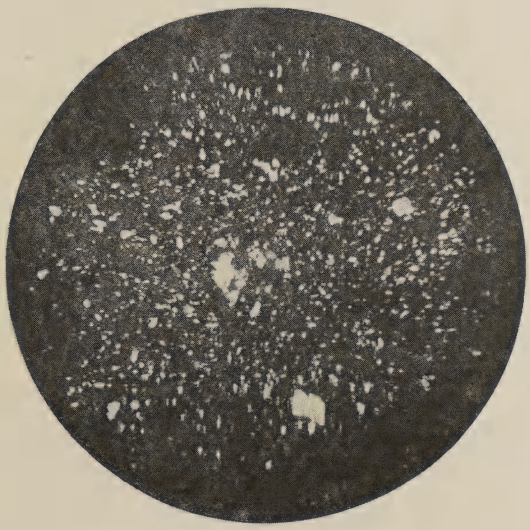


FIG 14b — Clay No. 239, Crossed Nicols.

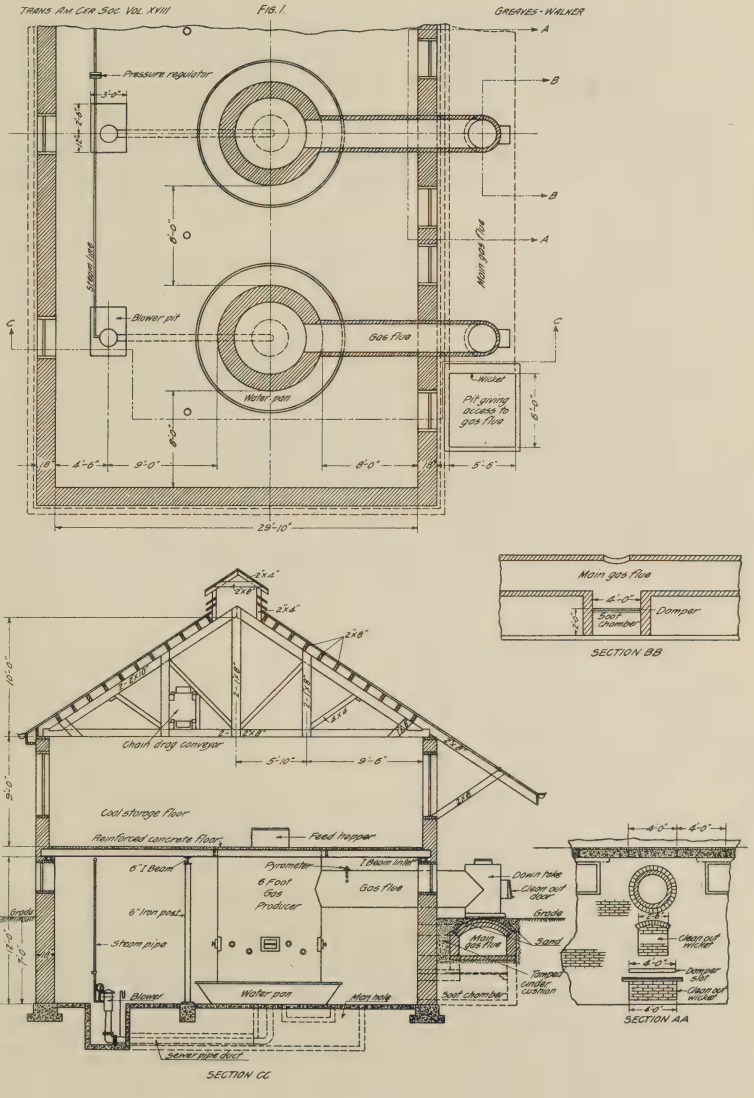
THE DESIGN AND CONSTRUCTION OF A PRODUCER-GAS HOUSE FOR CLAY PLANTS

BY A. F. GREAVES-WALKER

The use of producer gas in the clay industry is undoubtedly growing, although its growth is slow. In the last two years its successful use in connection with periodic kilns, both up and down draft, as well as with dryers and boilers, without the use of preheated air, has given it an added impetus. The continuous kiln, in connection with which it is best known, has also been greatly improved recently, and this, in itself, would tend to extend the use of producer gas. No matter what can be said against the use of producer gas in the ceramic industry, it has proved itself a money-saver when intelligently used, and this fact alone is bound to widen its use in time.

The first cost of a producer gas installation has always been comparatively heavy, and therefore there has been a tendency on the part of users to skimp in every way possible. The housing of the gas producers has suffered particularly in this respect. Poorly designed and constructed gas houses, however, are also due in part to a lack of knowledge on the subject. Even the ceramists who were best informed had many things to learn, and as is generally the case, the required knowledge could only be had from actual experience.

Originally, little thought was devoted to the gas house. As a general rule it consisted of a roof over the producers, a mere open shed. Little attention was paid to such details as the handling and storing of fuel, handling of ashes and the accessibility of flues. Naturally this added to the running expense of a plant. Conditions in our industry differ materially from those in the steel industry. In the latter, auxiliary producers, flues and furnaces are generally provided to take care of necessary shutdowns. In the clay industry this is not the case. Only such


TRANS AM CER SOC VOL XVIII
FIG. 1.
GRAHAM-WALKER
Pressure regulator
Steam line
Blower pit
Water pan
Gas flue
Main gas flue
Wicket
Pit giving access to gas flue
29'-10"
Main gas flue
Soot chamber
Damper
SECTION BB
Chain drag conveyer
Coal storage floor
Reinforced concrete floor
Feed hopper
6" I Beam
Pyrometer
I Beam lintel
6 Foot Gas Producer
Gas Flue
Down take
Clean out door
Grade
6" Iron pipe
Steam pipe
Blower
Water pan
Man hole
Soot chamber
Main gas flue
Sand
Tamped cinder cushion
Sewer pipe duct
SECTION CC
Clean out wicket
Damper slot
SECTION AA

equipment is provided, as a rule, as will keep the plant running, and if anything happens to it, a shut down becomes unavoidable.

Conditions cannot always remain so, and the time is approaching when the clayworker will learn that his gas plant requires as much attention as any other part of his equipment, if not a little more.

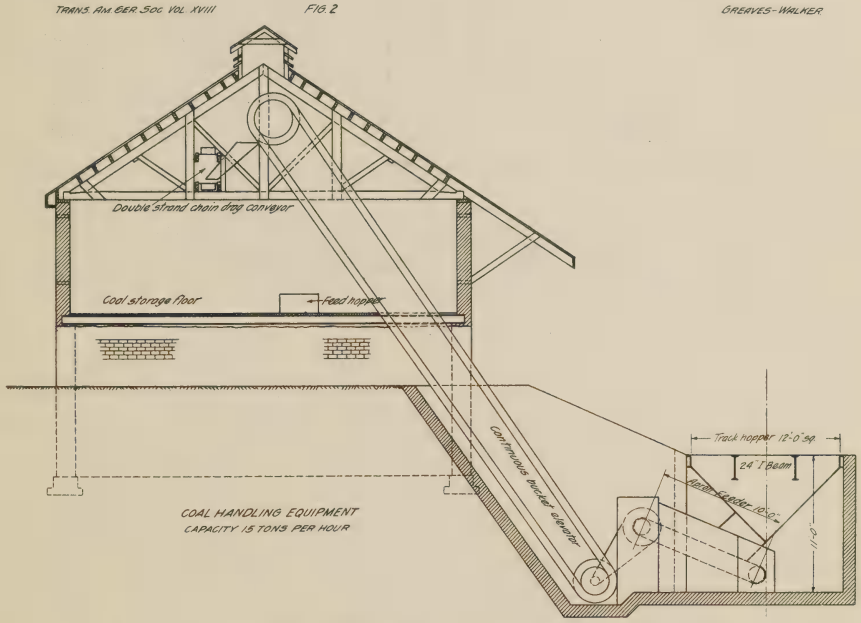
Design of the Gas House. In design, the gas house is quite simple. The building should be strong, roomy and have good ventilation and light.

If an extra producer is not installed with the original equipment, space should be allowed for one at the time of the erection of the building. The charging floor has to serve as a coal storage, and it is therefore necessary to so design it that it will readily carry from 25 to 50 tons to each producer. Many charging floors are built of heavy timbers and 2-inch flooring, but a steel and concrete floor is by far the safest and best, especially as it eliminates a fire hazard. Of course coal pockets could be built and a lighter floor put in, but this is an expense that is hardly justified on the average clay plant. Sufficient head room should be left above the charging floor to allow for swinging the long pokers. Roughly, this means that the roof should be the same distance above the tops of the producers as the producers are high. Plenty of large wall openings should be left on this floor, and a monitor should run the entire length of the roof. Ventilation is very important as gas fumes quickly sicken the producer men.

Laying out a good cleaning floor is important. In the first place sufficient room must be allowed all around each producer for handling bars and pokers in cleaning. This should not be less than eight feet. Space must also be left around each blower so that it is accessible from all sides. Blowers are often placed in the most inaccessible places, to the detriment of the operation of the plant. The water pans or water seals of the producers are often set so that the tops of the pans are flush with the floor. This is a mistake, as the pans are then much harder to clean. If set up on the floor, like a saucer, a man does not have to break his back to clean them, and the cleaning doors come

about on a line with his shoulders, the best possible position for barring down clinkers.

The wall openings on this floor should be near the ceiling, and there should be as many as possible, to provide not only the light necessary, but also ventilation since the temperature on this floor gets very high in warm weather. There are few hotter jobs than cleaning some producers, and if plenty of air is not provided, men cannot stand up to the work.



One of the drawbacks to the use of producer gas has been the necessity for burning out the flues every six to ten days. This generally means a ten to twenty-four hour shut-down — a clear loss. In seeking the reason for this, the writer found that practically all of the difficulty lay in the accumulation of soot and tar in the producer necks, and under the gas down-takes in the main flue. When there was scarcely any accumulation in the kiln flues, these points would be practically choked up, thus cutting off the gas supply. As no provision is ordinarily made

to clean out the flue under the down-takes, it is necessary to "burn out". By building openings into the main flue from the cleaning floor, this difficulty is entirely overcome, with the result that it is only necessary to burn out every thirty to sixty days. The method of construction of these clean out openings may be seen in Fig. 1.

Equipment of Blowers and Producers. In order to have proper control over the gas making, the producer house should be equipped with the proper instruments. Each blower should be equipped with a steam gauge inserted in the steam line, and a draft gauge inserted in the blast pipe between blower and producer. In each gas neck should be placed a pyrometer, so that the gas can be kept at the proper temperature. In the main steam line which supplies the blowers, a steam regulating valve should be placed in order to overcome fluctuations in boiler pressure.

When producers are equipped in this manner, it is an easy matter to train men to handle them and equally as easy to get perfect gas and perfect results from it.

DISCUSSIONS.[1]

(867)

[1] The order of discussion follows that of the original papers. See Table of Contents.

PRACTICAL NOTES UPON THE MANUFACTURE OF FIRE BRICK SHAPES.

R. H. H. Pierce: For some coal-gas installations, silica brick are surface ground by machinery to less than .01 inch variation. Such shapes are laid dry, and on heating, the expansion of the silica makes absolutely gas tight joints.

Regarding grogs, that produced by calcining flint fire-clay is the best.

In drying thin flat tile, a thickly sanded pallet is also effective as it allows the shape to "creep" freely. As a mold lubricant, the use of river sand, or clayey sand would not be permissible on high grade brick. I would emphasize the statement that the demand is for more intelligent preparation of materials now in use to meet individual conditions, rather than to discover new refractories.

R. J. Montgomery: In connection with a paper of this kind I believe it would be well to criticise the usual method of proportioning clays used in batches which are made into special shapes. A company will go to considerable expense to make molds and keep them in good condition, pay special attention to the molding, drying and burning but will still proportion their batches by the wheelbarrow load or at best use cars of a certain volume capacity. It is well known that the volume method of proportioning a batch is very crude and will never give a batch of uniform composition. The weight of a cubic foot of clay will vary greatly due to the moisture, fineness of the grinding and the amount of packing in the box. This variation is sufficient to seriously affect the quality of the shapes manufactured and cause defects, such as lowered refractoriness that do not appear until they fail in an installation. Shapes are usually made of especially selected clays and are of enough importance to justify the slight increase in cost of weighing the clays, making an allowance for moisture content when necessary. The weighing method of proportioning has been adopted by practically every industry even when large amounts of crude materials are handled.

H. L. Longenecker: I consider Mr. Montgomery's argument in favor of proportioning by weight extremely well taken. The reason this method has never been used, or at least not on any large scale, is undoubtedly because it has been considered an unnecessary expense. This refinement will come just as soon as fire brick consumers are in a position to enforce specifications which will make proportioning by weight a necessity. Many refractory manufacturers have, by more intelligent handling of their raw materials, within the last decade, surpassed the bounds of their own imagination in meeting specifications. We have every reason to believe that by the application of engineering methods to the manufacture of refractories a proportionate increase in quality will be made in the future.

CHEMICAL PORCELAIN

L. E. Barringer: I wish to call attention at this point to recent developed glasses, amongst which is that known in the market as Pyrex, for which much is claimed in the matter of heat resistance and also as to insulation value. In looking at Mr. Klein's slides this morning, I think many of us would obtain the impression that such a conglomerate mass might be expected to possess relatively poor insulation value and to be rather low in mechanical strength and that if the elements could be brought into a uniform homogeneous product, or mass throughout, this would accomplish the very best results. It might be said that glass represents the end to attain, and yet, if glass is a perfect product physically it is difficult to explain why we do not find the great insulation value that should be expected in Pyrex, or similar heat-resisting glasses. In the matter of disruptive strength Pyrex is apparently no better than porcelain, and yet the physical structure must be entirely different. In the matter of mechanical strength, Pyrex is very strong, and yet porcelain may be made just as strong and yet retain its crystalline structure. On the other hand, we have the very coarsest products showing quite remarkable mechanical strength as for instance some of the corundum and carborundum mixtures. I have been informed that the introduction of 5 percent or 6 percent of carborundum into sagger mixtures would increase the strength immensely, and if it were not for the high price, such mxtures would be used. I only cite this to raise the question as to the reasons why these various products act as described. The heat-resisting glasses are very high in boron, which is of interest in view of the fact that the papers seem to indicate that alumina is the essential feature.

A. V. Bleininger: It seems to me, from the points raised by Mr. Barringer, that it would not be altogether impossible to produce an artificial flux to replace feldspar. In this manner it might be feasible to accomplish things impossible with the use of feldspar. We might in fact, control the kind of glass ultimately produced in the final porcelain. Thus for instance, if

necessary, boric acid might be introduced in the synthetic flux and the maturing temperature of the porcelain lowered correspondingly.

Mr. Purdy stated that the Norton Company have attempted the preparation of a feldspar alumina mixture fused in an electric furnace, and so far have not been successful in its use. They have not lost faith because of the reported success by the German investigators, but so far have not found that that material, in any mixture tried, would decrease tendency to deform at high temperature, especially if there is any span or load.

Mr. Spurrier suggested that an etched piece of glass be examined under a microscope. He explained that while it may be visually homogeneous, it is not optically so, but shows a very nice development of crystals: they do not have any needle-like structure, and for that reason we would not expect any greater strength there than from the strength developed by the interlacing of sillimanite.

A CONTINUOUSLY OPERATED TUNNEL KILN FOR HIGH-GRADE WARE

Mr Burt noted that the continuous kiln at the Royal Porcelain Works, Berlin, was not mentioned among those having a movable firing zone. He also inquired whether the length of time during which the ware is exposed to the maximum temperature is left to the judgment of the burner, and how the movement of cars is regulated.

Mr. Barringer replied that the burner is nearly always in the neighborhood of the firing zone and by going to the top of the kiln, one can easily look down through the opening and observe the condition of the cones. The movement of the train through the kiln is not strictly continuous, but proceeds by steps, each step being a car length. The whole train of 36 cars is moved approximately every 2 hours. Just as soon as a car is pushed into the tunnel one car is pushed out of the firing zone and another pushed in. The burner knows that the next car is in the firing, or critical zone, and determines when it is to be taken out. When that car is taken out, another passes in and so on. The responsibility lies in looking after the car which is in the firing zone. The burner passes the word that he is ready for another car, and the time he keeps each car in the firing zone will sometimes be slightly under and sometimes slightly over 2 hours, but the period of two hours may be taken as an average. Cone 11 down is the maximum temperature of the kiln.

Replying to Mr. Montgomery, Mr. Cermak stated that there was no slope in the kiln track, and that two men can ordinarily move a train of 36 cars. The employment of gravity feed would introduce a difficulty in construction which would not be compensated for by the labor saved. No ware which is easily tipped over is set in this kiln.

Replying to Mr. Horning, he stated that asbestos packing one inch thick is placed between the cars to regulate circulation of gases.

Mr. Barringer stated regarding uniformity of product, that at first there was much "off colored" ware, but this was cor-

rected and at present the product is equal to, or superior in appearance, to that from the periodic kilns.

Replying to Mr. Potts as to the practicability of the kiln for drain tile and such wares, he said that for any ware that can be set on a car, and pushed thru on cars, this kiln would be practical and especially for ware not requiring particular care as to color and dimensions. The kiln has been more thoroly successful at Keasbey than at Schenectady altho they obtain excellent results in firing porcelain.

Of course there are considerations in a system of this kind which must be thought of before one builds the kiln, such considerations as losing the whole kiln system at once if any thing happens. It would always be necessary to be safe-guarded by having a certain number of periodic kilns, and I notice in all the German factories that they do not depend entirely upon the continuous system, but have a certain number of round potters' kilns. If a three or four-kiln plant attempted to operate entirely with a continuous kiln, there would be days of no output at all, and periodic kilns are necessary if you want to guard against differences in producton. The greatest possible good of a kiln of this type lies in a very large, continuous production the year round. If the production is erratic, that would be another consideration.

Replyng to Mr. Stover's question regarding fuel economy as compared with 18 ft. kilns, Mr. Barringer stated that at Keasbey an economy was proven, and that the Didier, March Company published a bulletin on this kiln which contains such information.

Replying to Mr. Watts' statement that the kilns at Altwasser have a pair of auxilary or pre-heating fire boxes about three car lengths ahead of the main firing zone, he stated that this was apparently necessitated by the higher temperature required. The kilns at Schenectady and Keasbey have only one firing zone with two fireboxes on each side of the kiln.

AN ATTEMPT TO FIT ENAMELS TO PLASTIC CLAY BODIES.

Mr. Parmelee asked if some of the crushing was not due to the grog in the body.

Mr. Kirkpatrick replied that he thought not, because there was no difference in results obtained with clay bodies and grogged bodies.

Mr. Parmelee said that when people are using a fire clay body they ordinarily add glue or gelatin to their slip in order to accomplish the results being sought here.

METHODS OF CALCINING CLAY FOR GROG.

R. R. Hice: The shaft kiln referred to by Professor Harrop is located in the Mount Savage district. In addition to this vertical kiln there are two others in use in Pennsylvania. These are located at Dean, Cambria County. These kilns are equipped with fire boxes, no coal being introduced with the clay. In practice the kilns are found satisfactory in so far as results are concerned, but the cost is practically the same as calcining clay in the regular brick kiln. As regards the character of the finished product there is practically no difference whether calcined in the common brick kiln or in the shaft kiln.

It is to be noted in this case that the shaft kilns are equipped with four fire boxes each, so that the labor cost is practically the same as with the down-draft kilns used for the same purpose, the only difference being that with the latter the action is intermittent, the kilns being filled, fired, cooled, and drawn, while with the shaft kilns the filling is continuous, the firing continuous and the drawing takes place also continuously. It would appear that the character of the clay very largely determines the possibility of using the shaft kiln for calcining purposes.

C. B. Harrop: The company operating the two shaft kilns in the Mount Savage district, state that they can calcine their run of mine flint clay much more economically in the larger down-draft periodic kilns. This same statement has also come, after years of experience with various methods of calcining, from one of the largest manufacturers of refractories in the Middle West.

METHODS FOR THE FACTORY TESTING OF PLASTER OF PARIS.

W. E. Emley: It is true that the determination of water is frequently merely a measure of the age of the sample, but if the plaster has been so exposed that it has taken up an excessive amount of combined water, its ability to set will be materially impaired.

The start of set is of great importance, because if plaster is mixed after its initial set has begun, the quality of the mold will be deteriorated. This start of set occurs before the temperature begins to rise, and cannot be read from the temperature curve.

The strength of the set plaster is one of its most important properties in the ceramics industry. It would therefore seem that its measurement should not be lightly passed over as of minor importance.

It might be well to lay more emphasis on the way plaster is is affected by minute amounts of various reagents. To obtain concordant results in the laboratory, it is absolutely essential that the dishes used be clean, especially free from all traces of set plaster: that the water be distilled, and the sand washed. The small amounts of impurities which may be introduced by neglect of these precautions will have a very marked affect upon the time of set and the strength of the plaster. Obviously, the same indeterminate factors may be expected to appear during the use of the plaster. The results of laboratory tests should therefore be interpreted with extreme caution, and are practically worthless unless a specified method of using the plaster is rigidly adhered to.

METHODS OF MAKING THERMAL CONDUCTIVITY TESTS, AND THE TRANSMISSION OF HEAT THROUGH BRICKS.

E. P. Poste: I think it may be of interest to say a word as to actual results. A year ago we were considering the installation of this very material in connection with a new furnace. Our old furnace, the temperature of which is maintained at 2000 degrees F., had a wall consisting of five nine-inch courses, and under ordinary conditions, the outside wall was very hot; if water would not actually boil on the surface, it would steam very noticeably. In building a new furnace we cut down from five courses of brick to three: inside course high grade fire brick, second course low grade fire brick, and an outside course of Nonpareil. If the outside temperature is an indication of the heat being lost, it is very apparent that we are losing much less heat by radiation on only a little over half of the wall thickness, it being possible to hold the hand continuously upon the outside surface of the new furnace while operating at the same temperature as the old. Aside from this we consider that we have more than saved the cost of the insulating brick by being able to substitute one course of brick where we had put in three courses. We did not place any in the crown of the furnace, fearing it would get too hot at that point. We were advised to put in only one inside course of fire brick instead of two. We were afraid of this however, as we depend to a considerable extent on the brick for heat storage and did not wish to cut this down too much. By erecting the furnace as above, using the eighteen inches of fire brick and nine inches of Nonpareil brick we produced very satisfactory results. The change involved stepping from a small furnace to a relatively large one with a re-arrangement of burners, so it is impossible to give actual data as to relative gas consumption.

FACTORS TO BE CONSIDERED IN PORCELAIN INSULATOR DESIGN.

L. E. Barringer: Mr. Ward has very clearly covered some of the troubles experienced with porcelain insulators, particularly line transmission insulators. If I understand the matter correctly, those troubles may be summed up as troubles which may result from (1), porcelain failing under sudden temperature changes; or, (2), under mechanical shocks; or, (3), the difference in the coefficient of expansion of porcelain and materials used in connection with it, these coefficients of expansion not being properly related. Troubles due to faulty design or use of improper supporting or accessory materials of course cannot be charged to the ceramic engineer, but must be left to the electrical and mechanical engineers. Insulators must be properly designed for the voltage, mechanical strains, climate, humidity, etc., and the materials used in assembling insulators must be such as will not cause failure. With the most perfect of ceramic products the best results will not be secured if the insulators are not properly designed and handled. It seems to come down, then, to making porcelain heat-resistant, resistant to sudden temperature changes and, above all, homogeneous. I have seen insulators taken from a line where there existed in the same piece porosity in one place and perfect vitrification in another. Such lack of homogeneity is fatal, and megger measurements will often show a great difference in insulators in the same line; some will be just as good after a year or two of weather exposure as originally; others will have fallen off tremendously in insulation resistance due to incomplete vitrification or a slow absorption of moisture through the glaze. There has been a great deal of discussion as to proper methods of testing, whether to use 60 cycle or high frequency, or both, and we have two electrical engineers here to-day: Mr. Creighton, of the General Electric Company, and Mr. Skinner, of the Westinghouse Co. I think this is a splendid opportunity for them to address the Society with reference to some of these problems.

E. E. F. Creighton: I should like to bring a message to this Society from the electrical engineers. This problem of porcelain insulators is one of the biggest ones we have before us at the present time. Insulators that two or three years ago were considered entirely satisfactory, have failed in thousands on the line. There is no solution in sight. Some improvements in insulators have been made, but it will probably be two years before the changes that have been made have been tried out. The several manufacturers of porcelain have recently decreased their selling price, and in so doing they have cut themselves out of spending enough money experimentally to get the solution of the problem. I worked on this problem first from the electrical side, and more recently have taken it up from the ceramic side, for the reason, which seems perfectly evident, that it is a combined problem of electrical and ceramic engineering. The ceramic engineers have not the facilities for getting the electrical tests that they need, and I can assure you that the electrical engineer is in no position at all to solve the ceramic problem.

The losses to the electrical transmission engineers today are something enormous. One engineer told me he spent $25,000 this last summer, changing over his insulators on one line. The selling price of an insulator is something like 90 cents for a single disc: it costs perhaps $25 to replace one of these insulators, taking into account the amount of testing that has to be done along the line to locate the faulty one. The loss of prestige is very much worse. Nearly all the interruptions of service on systems fed by long distance transmission lines are due to faulty porcelain insulators.

In regard to the matter of testing — my work has been mostly in the use of high frequencies and I hope that it will make it easier for ceramic engineers to take up the electrical side with this new apparatus. The apparatus known as an oscillator gives a frequency of about two hundred thousand cycles per second and is simple to operate. It is attached to the 110-volt circuit, and you do not have to have any complications with ammeters or volt-meters or watt-meters, or frequency-meters, or sine wave generators, etc. The one desirable measurement, voltage, is taken simply by sliding a little rod along, the way you

measure distance in inches. By that simplification it is possible to get rid of a great many rules that the electrical engineers have made, and the improvements that can be made in porcelain can be tested out by the ceramic engineer. This matter, if any of you are interested in it, is published in the *Proceedings of the Institute of Electrical Engineers.* I have a few extra copies left.

C. E. Skinner: I have thought for a good while, that we might so standardize tests on a lot of materials that we would bring about a better understanding and a better development of the fundamentals, and I have been trying to work along this line for a good many years. Among other things, a committee of the *A. S. T. M.* is trying to establish certain standard methods in connection with the testing of various insulating materials, including porcelain. It is the idea that if we can arrive at certain fundamental tests, all those who are making the tests will follow the same methods and the same standards, and thereby soon arrive at a better understanding of these things. Now this does not mean that in testing porcelain, for instance, that these standard methods will be all the tests that would have to be applied: but if we can establish, for instance, the high frequency test that has been mentioned, the flash-over test, and a few other fundamental tests, with an exactness such that two observers at different places or at different times will arrive at practically the same results for a given piece, we will have gone very far to establishing means of making progress along this testing line. I wish to emphasize, therefore, the desirability of establishing certain fundamental tests for all who have to do with development and testing work.

J. W. Ward: The comments that have been made in connection with the paper presented, bring out the fact that the problems to be considered cover questions of research, and investigation on the characteristics of the materials that make up the body of the porcelain, next, the question of a design that will be satisfactory for service, and last, a commercial test that will check the first two requirements and enable the manufacturer to turn out a product that will meet extreme operating conditions.

Experience so far with the insulating problems of transmission lines where the porcelain fails does not necessarily mean that in every case the insulator is at fault, in a good many cases the trouble has its foundation elsewhere, the question of distribution of stresses, the design of metal attachments, the protective devices, these points all must be considered and their relative values taken into account in the final analysis.

Porcelain insulators in the past few years are rapidly becoming a highly specialized product, and new standards are continually being advanced due to the rapid development of the electrical industry, and in order to keep up with this development it demands the serious attention of the combined efforts of both the ceramic and electrical engineers to better understand and solve the problems involved. As these new standards are established the manufacturer and consumer are gradually better understanding the technicalities of the porcelain art.

NOTE ON THE DETERMINATION OF SEVERAL DISSOCIATION POINTS.

W. E. Emley: The authors of this paper seem to have overlooked the fundamental principle which governs dissociation. Calcium carbonate has a certain definite dissociation pressure at any given temperature. This pressure is more or less completely counterbalanced by the partial pressure of the carbon dioxide in the surrounding atmosphere, and either dissociation or association will occur, depending upon which pressure is the greater. Moreover, the difference between the two pressures is one of the controlling factors which governs the speed of the reaction.

Referring to Johnson's article, with this principle in mind, we find that the dissociation pressure of calcium carbonate becomes equal to the partial pressure of carbon dioxide in ordinary air at about 600°. At temperatures slightly above 600°, we would therefore expect dissociation to occur very slowly, it is true, but completely if conditions are maintained constant until equilibrium is reached. The present article therefore corroborates Johnson's work.

The temperature obtained by Bleininger and Emley were lower because they used limestone instead of C. P. $CaCO_3$. The acidic impurities in the stone tended to drive out the carbon dioxide, in much the same way that sulphuric acid would at lower temperatures.

In the cement and ceramic industries, this effect of such acids as silica would tend to lower the temperature of dissociation. On the other hand, the partial pressure of the carbon dioxide in kiln gases is greater than in ordinary air, which would tend to raise the temperature. These two causes acting in opposite directions may nullify each other, or they may not. At least no conclusions can be drawn from the data in the present paper.

The same reasoning applies to the authors' discussion of calcium hydroxide: when properly interpreted, their results corroborate Johnson's. They differ from Hursh's results probably because Hursh did not slake his lime with a "decided excess"

of water, and may have had a different physical form of hydroxide to begin with.

It is a well known fact that gypsum will lose all of its water at 105°. The authors should have considered the rate of dissociation. The dissociation pressure of $CaSO_4 .2 H_2O$ is very high at this temperature, so that the first part of the reaction takes place quite rapidly. $CaSO_4 .\frac{1}{2} H_2O$ at 105° has a dissociation pressure only slightly above the partial pressure of the water vapor in ordinary air, so that this part of the reaction takes place very slowly. Indeed, if the air is nearly saturated, the partial pressure of the water vapor may be greater than the dissociation pressure of $CaSO_4 .\frac{1}{2} H_2O$, so that no decomposition will occur. That this time element was overlooked is evident from the authors' statement that they got no decomposition at 80°. By giving the system time to reach equilibrium, $CaSO_4 .2H_2O$ can easily be reduced to $CaSO_4 .\frac{1}{2} H_2O$ at 80° even under ordinary conditions. If especial precautions are taken to remove the water as fast as it is liberated, so that the partial pressure of the water vapor in the surrounding atmosphere is kept practically at zero, $CaSO_4 .2H_2O$ will eventually lose all of its water at 80°.

THE INADEQUACY OF STATIC PRESSURE DRAFT GAGES.

W. A. Hull: Prof. Harrop has given us an analytical explanation of the fact that the so-called draft gage is not generally useful to kiln burners. Many plants have draft gages but most of them have never found application in the actual burning of kilns. The draft gage connected to the stack or to the flue between kiln and stack does not give the kiln burner any consistent information, and Prof. Harrop's technical analysis of the agencies affecting the readings of draft gages confirms the conclusion of many kiln burners that they could not use such a device. The burner's best guide in the matter of damper adjustment is its effect. He has to adjust his damper so as to make his fires burn as he knows they must burn to make the right burning progress in the kiln, no matter what a draft gage may happen to be reading on that particular kiln at that particular time. What the burner knows as draft is not the thing that can be measured at the stack or at the flue to the stack but the thing that drives the air through his fuel bed. That force could be measured, by means of a draft gage with tube connection through the kiln wall at a height that would be fixed in relation to the position of fuel bed. If all the kilns on a plant were alike, draft gages so placed could be made useful in the regulation of draft both by the practical man and by the technical man. In many plants there is a tolerably complete assortment of different kinds of kilns with different sizes and shapes of furnaces, and on such plants, any sort of draft gage connected in any sort of way would tend to add to the existing confusion.

I am inclined to agree with Prof. Harrop that the most useful information to obtain in connection with the draft of any kiln is the rate of circulation, in other words, the number of pounds of flue gases going from the kiln to the stack per minute. So far, there is no practical way of obtaining that figure in ordinary every day burning. If Prof. Harrop's suggestion of obtaining flue-gas velocity by measuring the drop in pressure between two points in a flue, can be applied in a prac-

tical way to kilns, even though a diaphragm with a large orifice should have to be brought into play, a good step will be made toward placing the burning of ceramic wares on a scientific basis.

H. L. Longenecker: For several years operators of large steam power plants have been using draft gages in the manner that Mr. Harrop suggests. In fact there are several well known efficiency meters for steam boilers which operate on this principle. These efficiency meters generally make use of two differential gages of the inclined tube type, for convenience in securing comparative readings, mounting the two gage tubes parallel. One gage is connected to give a differential reading between the furnace and stack (or in the breeching, just inside the damper), the other gage gives a reading between the furnace and atmosphere. Thus when abnormal conditions exist within the boiler setting, such as an extremely thick fire, — damper closed too tight, — leaky baffling, etc., the liquid in the gages moves beyond extremes indicated by fixed pointers, the gage most affected indicating to the experienced operator, the probable cause of the trouble.

The combustion engineer, "Trouble Shooter," carries a single inclined tube differential gage. With this he takes readings between the furnace, both sides of the damper, the different passes of the boiler and the atmosphere. Using these readings comparatively also equalizes any errors due to variation in the static pressure. By practice these engineers have established the proper draft losses thru all types of boilers, with all settings for efficient operation. After taking such a series of readings your experienced combustion engineer is ready to proceed to the correction of any difficulties in an intelligent manner. Sometimes the damper is mis-set with reference to the outside controlling levers, — there are holes in the baffling, or the amount of opening left by the baffling at some point is too large or too small, — the tubes are sooted, — or too thick fires are being carried. But whatever it is that is interfering with efficient operation, the draft gage sometimes supplemented with a gas analysis apparatus will locate the fault and the remedy is generally apparent.

Why cannot the draft gage be similarly used for the more efficient operation of kilns? For fixed conditions of kind of ware and setting, for efficient operation, there must be a proper draft loss thru a kiln at each period of a burn. A series of draft readings over the floor of a kiln will indicate where the draft is weak or strong, and suggest the proper arrangement of the floor openings or flue system to secure an equal distribution of draft and heat over the kiln. A comparison of draft losses thru two kilns of equal size, of similar design and similarly set, one being of greater efficiency than the other, should suggest a method of bringing them to an equality.

The different designs of kilns, kinds of ware and different methods of setting, together with the variation of draft at different periods of a burn, make this use of the draft gage a problem which may have a different answer on every yard, but it is not in any sense a greater problem for any one yard than that solved by combustion engineers when they established the proper draft losses thru the different types of boilers, boiler settings, and firing methods for all of the boilers used in the United States. Above all it is an apparent means to an efficient end.

THE THEORY OF FLOTATION AND ITS RELATION TO THE DRESSING OF CLAYS

T. M. Bains, Jr.: I do not believe that there has ever been much done in flotation in regard to clay. A case came up several days ago where they had graphite in clays, and wanted to recover the graphite. It could be done very readily by the Wood flotation machine. With the clays, the only trouble I see would be that you would have to dilute your clay solution too much: you would have to add at least two or three times the amount of water to your pulverized clay for flotation.

Replying to Mr. Hice, I would say that the separation of zinc blende from mica would be possible, from what I know of it. A conductor generally floats: mica is a very poor conductor. Zinc blende sometimes is a good conductor and at other times it is not it depends on the amount of iron in it and yet you can float blende under certain conditions. Mica does not float, I know, because it occurs in ores, and we do not get any mica in our concentrates for sulphide ores, while graphite will float readily.

Replying to Mr. Orton's question whether rutile and zircon and the rarer minerals which occasionally occur in the kaolins would come off readily, I would say that they would not: they are non-conductors and will not under ordinary treatment. We may be able to get certain electrolytes that will raise them. If you can get any chemical action with rutile, you can float it. There seems to be a way of charging those minerals that float chemically. If you can do that you can float them. Some zinc blende is absolutely a non-conductor, yet you can float it beautifully under certain conditions. That is worth looking into.

Replying to Mr. Teetor's questions, I would say that Mr. Callow and a great many that know most about flotation, find in ores that the mineral generally is positively charged and that the tailings are negatively charged: the concentrates are positively charged and the air bubbles negatively charged and the oil generally negatively. Forcing air through canvas will give a frictional charge: that has been proven by Thompson and other elec-

trostatic men, it will assume a positive charge under certain conditions in the water: therefore the air bubble would attract the sulphide or graphite, and you would have a flotation but it would not be permanent. Where the oil comes in, is that the oil is negatively charged generally and first surrounds the valuable mineral, forms a thin film. If you increase your amount of oil above 1.5 percent, instead of flotation, the process reverses, and you will get oil and the valuable mineral, settling to the bottom, forming coagules: that means that the re-action of the positive sulphide or graphite to the negative air bubble is not strong enough to overcome the repulsion of the negative oil to the negative bubble, and over 1½ percent of oil ruins flotation. After you get above 10 percent, then the buoyancy of the oil will float the mineral, and you will have oil flotation by "buoyancy" which is not from froth flotation.

I have seen by adding the slightest amount of acid to zinc ore, it will float again. Certain zinc ores will not. Sometimes you just add a small percentage of alkali solution and it will seem to change the charge. The Bacon Institute has investigated that also very thoroughly. The Mullin Institute has investigated it also and they have proved that all the sulphides are positively charged when put in water. So there is a conflict of technical opinon which often occurs.

I find that you can charge air bubbles either positively or negatively. Here is a case: J. J. Thompson, of England, has taken up rain splashing against the metal roof which charges the air one way and the water the other way. Salt water splashing against the shore charges the air the opposite way. By adding the slightest salt to your water which you are agitating, you will change the polarity of the air bubble, so in that case the Mineral Separation machine which beats the air in with the propellers, may give a different charge from air coming through canvas. In one case you have a metallic beater, beating air in, and in the other case you have a non-conducting substance like brick or canvas through which the air comes, and nobody knows for sure what the polarity of the bubble is. This is all theory, and it is the only plausible theory. They have been accounting for these things by surface tension, and the only thing that ac-

counts for most of the phenomena is electrical. A lot more research work must be done before we know what happens.

I might say here that the addition of turpentine to a solution, and rosanalene and a few other chemicals will reverse the polarity of that bubble: as far as we can find out, change the polarity; so it is easy enough to change your charges on the sulphides, on the bubbles or on the graphite by either acids, alkaline solutions or by the different re-agents you add like turpentine. Amyl acetate is a good flotation agent. A good many flotation processes do not use oil at all, or acid, but generally use some frothing agent to create a great number of small bubbles. It is absolutely necessary to have an immense number of small bubbles, and you get that by adding amyl acetate or eucalyptus oil, or other material which reduces the surface tension and alows a great number of bubbles to form.

S. G. Burt: We sometimes have in our glaze mills this very frothing and I have attributed it to the possible presence of turpentine, and the problem arises how to get rid of it, because it is a great nuisance, and the only method by which I have ever been able to get rid of it was by the water spray that Mr. Bains speaks of. If it is an electrical proposition, why shouldn't there be some other way of getting rid of it? Mr. Bains suggests that by the addition of some reagent like iron sulphate, we might reverse that, as he finds that by the minute addition of some of these sulphates, frothing will cease in the flotation. The cure, however, might be worse than the disease.

Edw. Orton, Jr.: You can separate pyrites from clays without any trouble at all, but you have to crush your clays and add two or three tons of water to get the flotation.

H. Spurrier: It seems that in the flotation processes, even the kind of oil is a matter of considerable moment. Now a lot of experimental work has been done with agents other than oil, for instance, acetic acid has been used very successfully: and to that end it might be of enormous importance to this country to know that the crude pyroligenous acid carries a little wood-tar oil in it in addition to acetic acid and carries advantages in the application of this method that have not been secured in any other way. There have been in the last year in *The Metallurgical*

Industry, a couple of articles of considerable interest that it might be of profit to the clay men to read, although at first sight it would look as if clay is not very amenable to such a process, owing to the conditions of working. Still, a little resourceful work might be done that would pay very handsomely.

R. H. Pass: A few weeks ago some clay samples were sent in to be tested. I blunged them thoroughly, and while being blunged, a very persistent and dense froth formed, and in this were suspended the mineral and air bubbles, whereas the clay remained below. On testing the specimen, I found it contained a very large amount of alkaloid.

A METHOD OF TESTING THE CORROSIVE ACTION OF SLAGS ON FIRE BRICK

F. K. Pence: My observations along this line have been chiefly with reference to the corrosive action of frits upon the lining of a frit kiln. The vitrified lining has been found to have decidedly longer life than the porous refractory lining, although the chemical relation between frit and lining were similar in each case.

NOTES ON BARIUM GLASSES

R. L. Frink: The subject of barium glasses is particularly interesting to me as I have carried on some quite extended experiments in the use of barium carbonate as a constituent of the batch for the making of window glass while with the American Window Glass Company, such experiments being conducted in a practical way, the glasses being made in a full size operating tank holding about 400 tons. This experience leads me to believe that glasses made in small crucibles, or in quantities of a few pounds, do not have the same characteristics as do glasses when made in large pots or in tank furnaces operating under practical conditions.

The mass action which occurs during the melting period seems to give to the finished glass quite different physical properties when such melts are made in large quantities of several hundred pounds, or tons, than when they are made in small quantities and have a smaller or larger melting area of batch exposed to the temperature of the furnace, and this seems to be particularly true of barium glasses, for when melts were made rapidly and at high temperature, the bending moment, crushing and tensile strengths were quite different from those obtained where the melts were made over longer periods of time, and at lower temperatures. There was also considerable difference found in these properties when the glass was melted by direct fire impinging upon the batch than when the same was melted in pots, and completely protected from fire.

I have long advocated the use of barium to replace lead in the making of cut glass, or where it was desired to have a glass of high dispersion and luster, but results in this direction have not proven satisfactory, due to the fact that it makes the glass too hard to make the cutting and working of the glass economical.

It would be interesting to know if more than one test for solubility, the results of which are found in Table VI, were made upon any given sample, and how close an agreement was obtained as to solubility, for personally I have never been able to obtain any concordant result by employing the method described for

obtaining solubility factors. Also what was the character of the surface of the glass when examined microscopically.

A thorough investigation of the viscosity-temperature ratios might lead to some very valuable and interesting information which would be of practical importance to the glass manufacturer, if such experiments were made in a manner as would permit of the conclusions drawn therefrom being relatively or directly applied to practical conditions, for it is these ratios which many times have made a success or failure of mechanical devices, molds and methods of operating the same. However, again I question whether results obtained in the determination of these factors from specimens produced by small melts would give sufficiently accurate data whereby the manufacturer could rely upon or apply it to the conditions under which he melts his glass. Nevertheless even when made in small quantities as mentioned in Mr. Williams' paper, a comparison could be made which would without question be of value, and I would sgugest that his determinations in this respect be made by heating the glasses up to a temperature sufficiently high as to make the glass quite fluid, and then determine by some method the viscosity ratio at different temperatures when the glass was cooling. In this manner he would more clearly approach the conditions which exist in the practical manipulation of the glass, and the data will be more applicable to practical conditions.

If Mr. Williams did not stir his sample melts, or re-melt them, it would seem almost inevitable that he would obtain a glass which is not homogeneous, for the tendency of barium, when existing in excess of three percent, to produce cords, lamina, and a heterogeneous glass is very pronounced, particularly so where it is used alone and not in combination with lime free from magnesia, at least such has been my experience.

Mr. Williams' paper shows an enormous amount of work and application to the subject, and it is regrettable that he has not had more practical experience in the glass manufacture so that he could more accurately apply his methods to those conditions which would more nearly approach those found in commercial manufacture and processes. However, let us hope that his en-

deavor will receive the appreciation of those who are able to apply his results, and that his paper will induce others to carry on further experiments on a larger scale.

A. Silverman: The American Window Glass Company tried experiments in which they substituted barium carbonate for lime and found that under certain conditions the load of the glass could be doubled.

The factory experience has been that where barium is introduced beyond a few percent, the glass becomes cordy. Whether this is true of all barium glasses, such as sodium-barium silicates, is an important question. The price of barium offers another difficulty. There is such a demand for barium compounds that it is almost impossible to buy them at a reasonable price for glass manufacture. We can not use the sulphate very well and can not buy carbonate at a low price.

THE EFFECT OF VARIATION IN THE COMPOSITION OF GROUND COATS FOR SHEET-IRON ENAMELS

E. P. Poste: I want to call attention to the fact that Mr. Landrum's impact tests were made on these same little wash basins I mentioned: they were turned upside down and the hammer falling made a very definite deflection of the steel, and the chipping he got was produced by this deflection of the steel. I tried to devise a scheme for working enamel in the steel and I found that with a given enamel I could drop a weight, in many cases seven pounds, from as high as 60 inches, and I could get a blow which would not injure the enamel at all, striking directly on the enamel surface. I could take that same weight and drop it a foot, the blow being slightly out of perpendicular to the surface it was striking, and it would gouge right through to the steel.

In any case my impression was that if you did not get a deflection of the steel, you were judging the crushing strength of the enamel rather than its adherence under an impact which deflected the blow from the ware.

Mr. Danielson: When I started this work, the enameled plates were laid on the cast iron base of the machine. With this arrangement the sheet steel was bent and although the enamel did not chip on the upper side it did chip more or less on the under side, no matter how far the weight was dropped. Therefore a piece of hard tool steel was placed on the cast iron base and the enameled plates were laid on top of the tool steel. As the drop of the hammer was increased, the enamel really did chip and I do not believe it was due to the actual crushing of the enamel itself. The enamels which showed the greatest adhesion did not crush and did not chip with a slight blow. However the less adhesive enamels, those containing manganese for instance, broke away from the steel in large patches showing exceedingly poor adhesion although there were no signs of crushing in these cases either. Because of these observations I believed I obtained comparative results of the adhesive power and not the crushing strength of the enamels.

R. D. Landrum: The German people have known for the last five years just what cobalt does: only a quarter of one per-cent put in enamel will make it stick, and they have been worrying themselves for five years to find out why this takes place. We have proved definitely that that percent of cobalt does make the enamel stick to the iron, whether it is an alloy or reaches into the pores of the iron, or what not.

W. A. Denmead: In my own experience it has not been possible to compare results by tests on a flat sheet: indeed, I cannot compare it so well with enamels as with porcelain cubes. We at one time made a large number of porcelain cubes and attempted to find how many blows it would take to break the cubes of different material and found it was absolutely impossible to get any uniformity of results, because we would often get one piece of a volume mixture that we simply could not break down with the machine, and 50 others, burnt right in the same sagger would break with the first blow. It would seem that we should have a test piece shaped like Mr. Landrum's wash basin in which the fag, etc., would not have such a great effect.

Mr. Danielson said his experience corroborated Mr. Landrum's statement about cobalt toughening the enamel. He thought Mr. Denmead's tests on porcelain were crushing tests rather than tests of bonding power, and hence would not require any special shaped piece. Mr. Denmead claimed that in his tests, the deflection of the piece was also a factor.

H. F. Staley: In regard to the effect of cobalt, nickel, and manganese, this paper verifies, by a series of carefully planned and executed experiments, general factory practice which has been evolved from a vast number of unsystematic trials. Mr. Danielson's results indicate that, barring the question of increased cost, enamels could be improved by the use of more cobalt than the usual one-half of one percent.

The increased toughness due to the introduction of part of the flint into the enamel batch instead of into the frit is a point worthy of consideration by sheet steel enamelers. In many cast iron enameling plants, and in a very few sheet-steel plants, this practice is followed with good results. It is hard to understand the mental processes or technical principles underlying the ob-

jection of some sheet steel enamelers to considering the introduction of raw flint into the enamel batch of a ground coat when they are adding such substances as clay and "setting up" agents.

It should be noted that there are no cobalt or nickel oxides in the last four series of enamels, which deal with increase of silica, increase of calcium fluoride, decrease of boric oxide, and increase of antimony oxide. Since it is generally accepted among sheet steel enamelers, as stated by Mr. Danielson, that "a ground enamel free from cobalt and nickel oxides is unobtainable" at least part of the poor results obtained in these series may be due to the absence of these important oxides. These remarks would apply especially to those defects which were common to all the members of a given series. Of course in series J., where an attempt was made to get a white first-coat, cobalt and nickel oxides were not permissible.

The poor results obtained on increasing calcium fluoride in series G. confirms, in part, the opinion held by the writer for many years—that fluorspar is not a proper ingredient for ground coat enamels of any sort. I hold this opinion although I am fully aware that it is used in small quantities as an ingredient of ground coat enamels in several successful sheet steel enameling plants. It would be interesting to find the effect of eliminating the fluorspar from some of these ground coats, at the same time maintaining the fusibility by adding a sufficient quantity of some more reliable fluxing agent.

R. D. Landrum: I think Mr. Staley is right, absolutely, in regard to cast iron, but in regard to sheet steel, it would be better to take your enamel past the point where it is too fluid than to leave it under. I found in my practice, and it is also the general consensus of opinion among the enamellers, and I find that in various plants over the country they do have a very definite point for withdrawing their ground coat. They have found by rule of thumb method and practice, a very definite point: they get this by drawing out a hair and seeing that none of the silica particles can be found in it, and noticing the surface of the enamels and noticing just the moment the bubbles stop coming up: and it is surprising the definite point that is reached in the various plants.

THE CONSTITUTION AND MICROSTRUCTURE OF PORCELAIN.

A. A. Klein: Referring to Mr. Watts' question on the purity of the Maine feldspar, most of it was orthoclase with only a small amount of microcline and plagioclase. Somewhat over 5 percent quartz was present. We employed a fairly accurate method of determining the quartz content. It consisted in powdering up the grains, immersing them in a liquid whose index of refraction lay between that of quartz and that of orthoclase. A camera was attached to the microscope and a plane glass plate, to which tracing paper was fastened, was used instead of the regular ground glass plate. The image of the field was obtained on the paper and the grains traced. Owing to the fact that the refractive indices of the quartz are higher than that of the immersing liquid, whereas those of the feldspar are lower, the two minerals could be distinguished by this method. The grains on the tracing paper were cut out with a fine cutting edge and weighed. The errors involved in the determination, cutting and weighing, seemed to more or less counterbalance each other and an accuracy within a few per cent was generally obtained.

Regarding Prof. Orton's question concerning the obtaining of the lantern slides, these were prepared directly from the negatives. The negatives were obtained in the usual manner using the microscope and the photomicrogaphic camera with transmitted light.

Referring to Prof. Parmelee's question of the minimum temperature at which sillimanite is formed, we find a trace in ordinary hotel china which is burned at cone 10 or in the vicinity of 1250°. As to the possibility of determining the fit of a glaze on a body by petrographic means as Prof. Parmelee suggests, we have not investigated this at all except to note that there is a certain amount of optical strain in the glaze. We are of the opinion that the polarizing microscope could be employed advantageously in this problem.

A STUDY OF BRISTOL GLAZES COMPOUNDED ON THE NORM BASIS

J. B. Shaw: This paper is interesting as an illustration of the compounding of glazes by the use of norms, otherwise there seems to have been little of value obtained by the study. Granting that the expression of the ingredients of the glaze in the form of norms facilitates their investigation in a three component system, there is no reason why the same result can not be obtained by sub-dividing the glaze ingredients into three parts in any other proportion than norms, and since according to the work of Rankin and Wright, (*American Journal Science,* Vol. XXXIX January, 1915) and others there are eutectic mixtures of the ingredients in the glaze whose melting points are lower than those of the norms and which would therefore be formed in glaze before the norms, there seems to be no good reason for the use of the norms in preference to some other composition.

This work is along the same line as that of Purdy, Vol. XIV, p. 95, and would be subject to the same criticism as offered by Bleininger in the discussion of that paper, *viz.:* that we are not far enough advanced as yet to justify the assumption that any of these minerals exist in the glaze at any time.

COMPARATIVE MEANS OF MAKING POROSITY AND ABSORPTION MEASUREMENTS

H. N. Cox: I determined porosity by soaking 48 hours in a boiling tube: that was our test for getting absorption. All the porosity determinations I have ever reported were made on the basis of specific gravity, getting partial absorption and getting suspended weights and weights in the air, the difference being the exterior volume. Figuring the porosity on that basis, the difference between the volume as figured from the dry weight and the true specific gravity and suspended and dry weight would be the volume of the pores both open and closed. I did not attempt to determine porosity by the absorption of water, but used that method for getting the open pores.

E. E. F. Creighton: In our electrical work we have to fall back on porosity tests, and I think the porosities we have to measure are so much smaller that they can come in a different category than suggested by Mr. Walker. We found with our test that it was possible with the vacuum test to get 100 percent difference in the amount of the water absorbed. The weight of water absorbed was on the order of a hundredth of one percent up to one-tenth of one percent. We have had insulators brought in from the line that have been out for a couple of years and have absorbed moisture from the cement: those insulators would have a reading from 100 megohms to 1000 megohms, indicating that they had absorbed some conducting material in place of water. One of those insulators was dried out and its insulation resistance went to infinity. It was then soaked in water for four weeks, — that is up to the present time, — and its resistance still remains infinity, showing that the absorption of moisture was extremely slow.

We had to go into those details because it is not permissible to have one hundredth of one percent porosity in electrical porcelain. It must be much more perfect than that. I think a description of the difficulties I had in making the test might be of interest. We took a piece as large as 250 grams, and as we did not care to break it, because that would introduce some small

cracks, possibly, we cut it out with a carborundum wheel, and all surfaces were carefully smoothed off. Then, in order to get the water to enter, it is necessary to do more than simply immerse it in water and evacuate it. We had first to evacuate it down to a few microns and hold this for several hours. Then without breaking the vacuum, introduce some thoroly boiled water in on top of the porcelain. It has to stand at least eight hours before the water can get into the pores. It is only after such a test that you can get any degree of thoroughness at all. The greatest difficulty comes after that: making the first weighing. It is easy enough to have the surface perfectly dry, but weighing after immersion, the question arises how much moisture is left on the surface. Since we are measuring down to a very small weight, the weight of surface moisture becomes very appreciable, and in order to eliminate that effect, we took readings over a certain length of time, in dry air. One can note the weight decreasing from minute to minute. It will follow along a logarithmic curve and when the curve becomes flat, one is as near as possible to the true weight of the porcelain plus the water inside, without the error due to surface moisture.

W. A. Denmead: I wish to back up Mr. Creighton's statement wholly. I will describe the method of the test in our own office: An insulator was broken, a piece approximately a cubic inch in volume was ground from the piece, all glaze removed and great care taken to remove any grease or anything of the kind. Upon 48 hours absorption in cold water, I have found many pieces, that on an analytical balance, sensitive to one-tenth of a milligram, showed no absorption.

I should like to explain regarding the porcelain absorbing less than 1/100 of 1 percent. We do have large quantities of porcelain absorbing less than 1/100 of 1 percent tested by the 48 hour immersion in cold water but not by Mr. Creighton's method. It is very often the case that porcelain showing the low absorption of the 48 hour test will show absorption in excess of 1/10 percent by Mr. Creighton's method, and there is a good deal of evidence to show that an absorption of this amount is very detrimental to certain types of insulators. I have added

this note as I do not find any evidence that 1/100 percent absorption by Mr. Creighton's method is fatal to an insulator.

I do not know whether any records have been made by the Society in the past, but on the Pacific Coast I found that it was quite possible to get an absorption determination much higher than that obtained either by boiling or immersion in cold water, by an alternation of each. It must be transferred from the hot water to the cold water immediately and back.

A. V. Bleininger: It is interesting to note in connection with this paper that Committee C-3 of the American Society for Testing Materials has recommended boiling for four hours in the absorption test of half bricks as stated in their directions for testing building bricks. The boiling method has the advantage of simplicity over the absorption in vacuum.

F. K. Pence: Before adopting a method of determining the absorptions given in the tables of my paper on "Classification of Floor Tiles as Related to Degree of Vitrification," Vol. XVII, I made comparative determination between the 3 hour boiling method and the method of 1 hour boiling followed by 24 hours soaking. I found the latter gave higher absorption and appeared to be preferable over the former method.

After reading the above paper I have been interested in making further comparisons as given in tables herewith.

The tiles used were 6 in. by 6 in. by ½ in.

The types selected were red semi-vitreous and buff semi-vitreous floor tiles, also white wall tiles bisque. Six tiles of each were used.

Absorptions were obtained on the basis of dry weight.

As will be noted, check determinations were not made on the boiling methods as this is given in the above paper. Also check determinations are omitted on 2 hours boiling and 24 hours soaking, check determinations being made on the two extremes of the composite method.

Tiles were immersed on edge in distilled water in all determinations.

Results were obtained as shown in Tables I, II and III:

TABLE I — RED SEMI-VITREOUS

TRIAL NO.	1	2	3	4	5	6	AVERAGE
3 Hours' Boiling.....	5.21	4.61	4.59	4.27	4.71	4.61	4.67
5 Hours' Boiling.....	5.41	4.71	4.93	4.49	4.97	4.72	4.87
1 Hour's Boiling } Det. A.	5.38	4.81	4.99	4.51	4.81	4.75	4.88
24 Hours' Soaking } Det. B.	5.35	4.74	4.92	4.38	5.02	4.64	4.84
Difference	.03	.07	.07	.13	.21	.11	.04
Average	5.37	4.78	4.96	4.45	4.92	4.70	4.86
2 Hours' Boiling } 24 Hours' Soaking }	5.49	4.79	5.08	4.43	5.04	4.67	4.92
2 Hours' Boiling } Det. A.	5.44	4.79	5.09	4.33	4.97	4.60	4.87
48 Hours' Soaking } Det. B.	5.56	4.94	5.15	4.54	5.15	4.79	5.02
Difference	.12	.15	.06	.21	.18	.19	.15
Average	5.50	4.87	5.12	4.44	5.06	4.70	4.95

TABLE II — BUFF SEMI-VITREOUS

TRIAL NO.	1	2	3	4	5	6	AVERAGE
3 Hours' Boiling.....	5.50	6.36	6.11	6.35	7.46	4.75	6.09
5 Hours' Boiling.....	5.69	6.85	6.49	6.73	7.77	4.82	6.39
1 Hour's Boiling } Det. A.	5.64	6.74	6.41	6.72	7.67	4.77	6.33
24 Hours' Soaking } Det. B.	5.51	6.69	6.49	6.75	7.84	4.70	6.33
Difference	.13	.05	.08	.03	.17	.07	.00
Average	5.58	6.72	6.45	6.74	7.76	4.74	6.33
2 Hours' Boiling } 24 Hours' Soaking }	5.78	6.98	6.64	6.85	7.77	4.77	6.47
2 Hours' Boiling } Det. A.	5.66	7.01	6.64	6.85	7.77	4.61	6.42
48 Hours' Soaking } Det. B.	5.85	6.95	6.60	6.82	7.84	4.73	6.47
Difference	.19	.06	.04	.03	.07	.12	.05
Average	5.76	6.98	6.62	6.84	7.81	4.67	6.45

TABLE III — WHITE WALL TILE BISQUE

TRIAL NO.	1	2	3	4	5	6	AVERAGE
3 Hours' Boiling.....	13.64	13.90	13.69	13.63	13.99	13.73	13.76
5 Hours' Boiling.....	13.86	13.96	13.96	13.94	14.33	14.10	14.03
1 Hour's Boiling ⎫ Det. A.	13.88	14.21	14.06	13.88	14.31	14.02	14.06
24 Hours' Soaking ⎭ Det. B.	14.03	14.25	14.17	13.97	14.41	14.08	14.15
Difference	.15	.04	.11	.09	.10	.06	.09
Average	13.96	14.23	14.12	13.93	14.36	14.05	14.11
2 Hours' Boiling ⎫ 24 Hours' Soaking ⎭	13.99	14.29	14.16	13.94	14.33	14.10	14.14
2 Hours' Boiling ⎫ Det. A.	13.98	14.29	14.16	13.96	14.35	14.08	14.14
48 Hours' Soaking ⎭ Det. B.	14.03	14.33	14.22	13.97	14.40	14.10	14.18
Difference	.05	.04	.06	.01	.05	.02	.04
Average	14.01	14.31	14.19	13.97	14.38	14.09	14.16

It will be noted that practically the same degree of difference is obtained between 3 hours boiling and 5 hours boiling as was obtained by Mr. Walker in his porosity determinations. Also that the 1 hour boiling and 24 hours soaking is equivalent to 5 hours boiling, and that the check determinations compare favorably with Mr. Walker's check results on the 5 hours boiling.

A slight increase in absorption is shown with 2 hours boiling and 24 hours soaking, and no material further increase is shown with 2 hours boiling and 48 hours soaking.

On the basis of checking results, the 5 hours boiling, 1 hour boiling and 24 hours soaking, and 2 hours boiling and 48 hours soaking, appear to fare equally well.

The method of 1 hour boiling and 24 hours soaking is convenient in operation and regular in result, although because of the somewhat higher figure obtained the method of 2 hours boiling and 24 or 48 hours soaking might be preferred.

CLAY AND SHALE RESOURCES IN THE VICINITY OF CLEVELAND, OHIO

Mr. Richardson inquired whether there was in the Cuyahoga Valley, or higher up in the hills, another formation above the Cuyahoga shale which overlays the Berea grit, and which was referred to as Orangeville.

Mr. Van Horn repled that another shale formation outcrops in the valley above the falls at Brecksville. Replying further he said that the brick plants on the lake shore have still 500 to 800 feet in Chagrin shale below their present operations. The total of the Ohio shales, formerly called Huron, Chagrin, and Cleveland, aggregate a thickness of 1,000 to 1,200 feet and the Huron shale comes to the surface in the Huron River.

Prof. Orton expressed skepticism about the red color of Bedford shale being due to an excess of iron, as he had been unable to get enough extra iron from them to account for their color.

Mr. Van Horn thought that analysis of the red and blue variety of Bedford shale differ probably only in the percentage or proportion of ferrous to ferric iron.

THE UTILIZATION OF OHIO CLAY-BONDED SANDSTONES FOR SILICA BRICK MANUFACTURE

W. Stout: The Clarion sandstone is somewhat friable for brick as the clay content is low, but the Gephart sandstone makes a brick very readily. The sandstones were crushed through a jaw crusher set at ⅜ inch size. This gave a material one-third coarse and one-third intermediate, and about a third that would pass a 20 mesh sieve. This was wet with water, allowed to stand over night, and hand-molded in the ordinary way. Here is a piece of the rock taken from the hearth of Jefferson furnace after one year's service. The dark layer on the edge is slag and is not over a sixteenth of an inch at the most. The next layer is about ⅜ of an inch in thickness, and is the part that Dr. McCaughey showed was practically all tridymite. It had been changed by the heat to tridymite and in so doing has expanded: so you see the porous spaces are comparatively small compared to those in the original rock. Beyond this the rock shows practically no effect of alteration.

Regarding the blue color where the slag has not penetrated, I think that is probably due to the physical state of the material. There is no difference in the iron content. Under the microscope it shows simply as tridymite with low polarization colors. You can not tell whether the color is due to the change of the material passing from quartz to tridymite or whether there is also a small amount of iron present. Under the microscope, however, you see no iron. The rock appears to be quite pure.

A. V. Bleininger: I think it is necessary to point out that a comparison of chemical analyses, burning tests carried only to cone 12 when the temperature of the average silica brick burn corresponds to cone 18 and softening point determinations do not suffice to establish the usefulness of materials for the manufacture of silica refractories. The matter is not as simple as that. The volume change upon carrying the bricks to cone 18, subsequent changes upon refiring, the resistance to sudden heating and cooling, the completeness of the cristobalite transformation and the load carrying ability at about 1400°C., are facts that

must be known before an intelligent opinion can be given. Thus a material may result in an apparently sound brick, but under the conditions of use it might go to pieces completely. Special caution is necessary in the case of clay-bonded silica brick which in American practice have not proven very successful. Some of the analyses quoted in this paper are of doubtful value, such as that of the Crummerdorf quartz shale showing an alkali content of 5.30 percent.

SOFTENING POINTS OF POTASH FELDSPAR—STEATITE MIXTURES

F. C. Stover: I found that by substituting in a mixture that called for one percent of whiting as a flux, about half whiting and half steatite I could get a good deal closer and tighter porcelain, and especially the lower I went in flint. I would even get down to zero flint allowing the rest of the mixture to be about the same; and while I haven't anything that would be in direct comparison with the gentleman's crucible, the trial pieces I did make would stand going up to white heat and dipping into ice water and would not fly at all.

A. V. Bleininger: In answer to Mr. Purdy's question what chemical porcelain The Bureau of Industry had in mind when they said that the Bureau of Standards had developed a chemical porcelain that beats the European, I might say that we have made a considerable amount of chemical porcelain which has stood very severe tests: in fact there were systematic tests conducted along this line in comparison with European porcelain which show that there is no difference at all. If difference were shown, I think it would be in favor of the material produced in the laboratory.

Mr. Staley notes that cryolite is mentioned as a cheap flux and states that it now costs seven and one-half cents per pound, and is never less than six cents per pound hence can not be considered cheap.

Mr. Montgomery replies that he is first looking for technical results rather than the cheapness of the materials.

Mr. Staley points out that fluorspar, even when only one-half percent is used will greatly lower the viscosity of a slag.

Mr. Orton reports that he has looked into the slag brick question and that a large amount of pavement work has been done. The imported slag brick used in Washington streets came from Middlesbrough, England. They are poured while very fluid, and later are annealed in large sand pits. They are astonishingly tough, being far better than any paving brick which he has ever tested. He could not see that the process was an involved one, and thought our metallurgists should apply themselves to the utilization of natural slags, rather than to producing a slag of synthetically low melting point.

Mr. Montgomery explained that iron slags are not suitable for making slag brick, and that copper slags only have been successfully used. The iron furnace slags are capable of being made into Portland cement, and should be thus used rather than for making slag brick.

J. S. Unger: I just want to verify Prof. Orton's statement that at Middlesbrough, England, they are making slag brick and have been making them for a number of years. They are made from blast furnace slag which differs from the American slag to quite a considerable degree chemically. They take the liquid slag, pour it into a mold which is made in two halves: as soon as it is solidified sufficiently to remove without breaking, they open the mold and throw these red-hot bricks into kilns. After the kiln is full, it is closed and the bricks are allowed to anneal from their own residual heat. Experiments have been made in the United States, attempting to do the same thing they have done in England, but the blast furnace slag we usually

produce here is of an entirely different character, being more glass-like, while the English slag resembles a stone: and in attempting to do the same thing, we have found that the slags made here are inclined to be very brittle. I know there are a number of investigators experimenting with American blast-furnace slag, hoping to add inert material which will change the glass-like character of the American slag. The silica in the English slag is about 26 percent, the alumina, about 22 percent: the average slag in the United States will run about 34 percent silica and 14 percent alumina.

Mr. Montgomery inquired how the inert material was to be added, and to this Mr. Unger replied that the proposed process was secret, but that the promoters expect either to add an inert material to the slag as it runs from the furnace, or to add a material which acts mechanically. In no case do they propose to re-fuse the slag as the cost of the brick thus produced would be too expensive.

In reply to Prof. Orton's inquiry as to the present utilization of slag, Mr. Unger said that assuming that this year we make 33,000,000 tons of pig iron, we will make about half as many tons of slag. A large amount of slag is used in the manufacture of Portland cement, in making railroad ballast, and in making material as a substitute for fine gravel in roofing work. A little is used in the granulated form in making what we call slag lime brick. However, the supply is many times in excess of the demand.

A MEANS FOR THE DETERMINATION OF THE FINENESS OF RAW MATERIALS

W. E. Segsworth: Some months ago Mr. Worth, of the Pennsylvania Feldspar Company, asked me to do some work in connection with his grinding mills, and naturally the first question I asked him was how fine the material was to be ground. He answered me, as I suppose a great many grinders would: "Grind to leave so much residue on a 140 mesh sieve." I said, "What about the fineness of the material below that?" He said, "If you get that residue the finer material will take care of itself." I was not quite satisfied with that and tried to get some further information on the subject, and in trying to get it I found that there was no standard whatever; and, writing to his customers, I found that some tested it one way and some another. It occurred to me that there ought to be some way for testing materials so that we could get results we could compare, so that if we write to our customers, the customers can tell us what they want and we can tell them what we are ready to give them; and if we cannot give them what they want, we can try to make it for them. About the first thing I discovered was that the elutriation process was used for grading the fine material which was too fine to be graded by sieves.

I did not know much about it, — though I found that a good many were using the method; but Mr. Walker seems to have discarded it altogether. He makes a curve and determines two or three points on it, and from that he infers the rest of the curve. I suppose that, if one could find an equation for the curve and you found two or three points on it, then you could find the rest of the curve from these points; but in order that the curve should have the same equation, you would have to have the material ground by the same process. I quite admit that if all the material was ground by precisely the same process, by getting two or three points on the curve, you could derive the rest of the curve from them; but it seems to me that if we are going to adopt any method at all we ought to adopt one more scientific than that. It may be that we will want to produce

the material by some other process. What we want to find out is what we want to make, not defining what we want to make by the way we make it: we want to define what we are going to make, first of all. I would like to ask Mr. Walker a question as to how he grades material below the finest sieve he uses? I notice from his diagram, that he merely has a dotted line beyond the sieves and I would like to know if he has any other method of finding out what the fineness of this fine material is?

Mr. Walker replied that the points plotted on the curve were sufficient to give him a good idea of what the remainder of the curve would be.

Mr. Orton asked what objection has been found with the elutriation method of classifying very fine material.

Mr. Walker replied that it presented difficulties due to the water used, to deposition on the sides of the cans, and to a tendency to get retarded flow between cans which causes a variation in the percentage of material found in different cans.

Mr. Watts stated that he had used a one can elutriator with success for classifying fine materials, but that the tendency for scum to form on the sides was noted, and doubtless retarded the action of the water slightly.

Mr. Segsworth suggested using glass elutriator jars.

C. H. Kerr: Fineness has been quite generally recognized as one of the most important variables in ceramic manufacture, but in spite of all discussion and agitation of the question it seems quite certain that the tremendous importance is not yet fully recognized. All reactions are tremendously influenced by the fineness of the reacting materials, and there is no question that the periodic variations in factory results are due, to a much greater extent than has been heretofore realized, to variation in fineness of one or more raw materials.

This paper adds very considerably to the preliminary work which must be necessarily gone through before satisfactory standards in testing and in factory practice can be attained. The 280-mesh sieve adds considerably to the information that we have previously gotten from sieves as fine as the 200, but it still falls far short of giving us much definite information of the actual

size since the important size comparisons are in the still finer grains.

Some modification of the elutriation apparatus must be brought to our assistance to accomplish a proper subdivision. It is desirable that the specific gravity of the separating medium be comparatively near to that of the suspended particles, but at the same time viscosity should be low. Probably a study of this specific gravity-viscosity problem in connection with elutriation would lead to considerable improvement. In any case, it is very certain that one of the real problems of ceramic manufacture is in the correct determination and regulation of size of grain of the raw materials.

THE EFFECT OF NON-PLASTIC MATERIALS ON THE SHRINKAGE AND MECHANICAL STRENGTH OF A NO. 3 FIRE-CLAY BODY.

C. H. Kerr: A criticism of this paper which can be applied to a very large number of researches upon which reports are constantly being made, is in the insufficiency in the number of test pieces considered. In practically all work with clays, especially such work as is concerned with strength determinations, it is unsafe to base conclusions on the average of any small number of test pieces. For such work it is rarely satisfactory to average less than ten individual determinations and in the majority of cases it is probable that more truth could be learned by still further increasing the number of individual determinations taken in the arriving at a given average, even if it be necessary to reduce the total number of points covered in the research.

It seems a doubtful assumption to attribute to the inversion and reversion of the different crystal modifications of silica as much of the cracking trouble as at present is being attributed to that cause. There are many very tender bodies with much less than 10 percent quartz, and while unquestionably the volume changes due to the modification of the crystalline form of the SiO_2 is a very vital ceramic phenomenon, the fact must not be lost sight of that it is unsafe to reason that these crystalline modifications are causing the trouble simply because the silica or quartz is present. Other physical changes connected with the peculiarities of the plastic body are often of considerably more importance.

Strength tests made on cold bodies are not necessarily a good index of the resistance of that body to cracking during the process of manufacture. The cracking phenomenon is always the result of many opposing forces, and the strength test of the cold body has not been shown to have a direct relation even to the strength of the body at the different temperatures attained in manufacturing. It is undoubtedly very misleading in many instances to draw conclusions regarding the behavior

of bodies while hot from determinations made on the body in the cold state.

R. J. Montgomery: I had a somewhat similar experience in the use of sand with fire clay. It was desired to use sand as a standard non-plastic in testing the grog carrying capacity of fire clays. We found that the strength, both in the raw and burned state was considerably lower than that obtained when calcined clay was used as grog. I believe the low strength was due largely to the smoothness and lack of porosity of the sand. The clay could not penetrate or get a hold on the round sand grain. Also the sand increased the refractoriness of the mix and required a higher temperature to obtain the same degree of vitrification.

THE MECHANICAL STRENGTH OF FIRE-CLAY BODIES AFTER REPEATED HEATING.

C. H. Kerr: The testing of the effects of repeated firing on a fire-clay or other refractory body has unfortunately received about as scant attention as has been given to the importance of the resistance of the body to the repeated heatings and coolings. It is unquestionably a phase of the problem which has been about the most neglected of all. For saggers it seems probable that testing of the cold body between successive heatings will disclose to a very large extent the comparative values of different bodies and their ability to withstand such treatment. However, in the case of such crucibles, glass pots, etc., as are subjected to a considerable temperature variation during the process of manufacture, but in which they are not cooled to atmospheric temperature or anything like that temperature, it is possible that in some cases tests made on cold bodies between successive firings might be quite misleading. With clays of widely differings chemical and mineralogical composition the chances of error in making comparisons in the cold state are much greater than with clays of the same general type. With siliceous materials this possible discrepancy is probably of very considerable importance.

A. V. Bleininger: Mr. Kirkpatrick complains that there is lack of relation between the porosity and the mechanical strength as measured by the modulus of rupture in his repeatedly burned pieces. It seems to me that he can hardly expect to find any such correlation inasmuch as the decided loss in strength is evidently associated with some molecular change in the clay, such as the formation of sillimanite in either the amorphous or crystalline form. Mr. Kirkpatrick's most frequently fired test pieces were examined somewhat hastily by one of our petrographers, Mr. Brady, who stated that there was no evidence of crystalline sillimanite. Whether the true specific gravity will indicate the structural changes involved is problematical. There is some danger of drawing erroneous conclusions from this work. It deals, as the title implies, with the determination of the

mechanical strength of a number of clays after repeated firings, no more, no less. Consequently we should be somewhat cautious in applying these conclusions to a sagger or anything else. The results of the work certainly are interesting and give us numerical values showing the real loss in strength suffered by clays upon reburning. At the same time they indicate in an indirect way that better results are to be expected from mixtures of clays than with individual clays.

F. A. Kirkpatrick: Since the writing of this paper, further data has been obtained, of which the following is a summary:

True specific gravity changed during the series of burns only 0.01 to 0.02 for each body except New Jersey fire clay B, which suffered a decrease of 0.05 from burn 2 to burn 7. The general tendency was toward a very slight decrease. Enclosed pore space averaged 2 to 4 percent. Open and total pore space decreased slightly from burn 1 to burn 4 and remained practically constant thereafter.

The modulus of rupture tests are purely physical tests and the results do indicate the rate of change in strength of fire clay bodies which are subjected to repeated heating.

The causes of the deterioration of the bodies have not been determined. The true specific gravity changes are confusing. Microscopic examinations may or may not throw light on the question. The causes are several and probably include mechanical stresses due to structure as well as molecular changes.

VARIATION IN LINEAR SHRINKAGE OF CLAY TEST PIECES

Mr. Purdy inquires as to the variation noted in testing molded or wedged clay. Mr. Bailey replies that if the clay is made to fit the mold before being placed in it, the results are very good. If the clay is smaller in diameter than the mold, but longer, and is forced to take the form of the mold by pressure, there is a noticeable variation in shrinkage. If the clay mass is very large, however, the difference in its shrinkage in different directions does not differ greatly.

Mr. Parmelee inquires as to variation in cast trials.

Mr. Bailey replies that such trials were very unsatisfactory. The linear shrinkage was too delicate in such trials and the variation was too large to distinguish between satisfactory and unsatisfactory clays. The volume change was used exclusively in comparing cost trials.

When the clay sticks to the mold, it drys out and will stretch the clay in whatever direction it sticks, and that will have an effect on the burning and the drying shrinkage too: we do not do very much work in the plaster of Paris molds. What I have done I have found very good. We test the clay by a tensile test machine and make the brickettes in a brass mold, and by making the clay in the shape of the specimen before it goes in, I have gotten very good results that way. Little benefit has been noticed in the clay which has been subjected to the long ageing treatment. The greatest improvement is noticed during the first week. We age our clay about three or four weeks.

C. W. Parmelee: In considering this phenomena it is well to remember that pressure applied to the day mass does not cause the colloidal mass to flow out from between the particles since the total amount of colloidal material is so exceedingly small that the movement of the clay mass cannot be thus explained. What happens is the flowing of the smaller granular particles which are coated with the exceedingly thin film of colloid, which movement is also facilitated by the presence of water.

The testing of shrinkage of clays by preparing a slip and pouring into shallow plaster of Paris molds which are intended to deliver discs 10 cm. diameter by 1 cm. thick is used more or less widely. The method is very attractive because of certain evident advantages, and has been recommended for use. However we have had such alarming variations that it has been abandoned. It has been our experience that the amount of shrinkage on the pieces so prepared varied according (1) to the "hardness" of the mold, i. e. the proportions of plaster and water used in its making: (2) the dryness of the mold: (3) as Mr. Bailey suggests, the condition of the face of the mold, i. e. whether rough due to much use.

J. Bailey: In concluding I wish to state that we have made no systematic tests with cast specimens.

Mr. Parmelee's statement that the colloidal material is too small to explain the movement of the clay mass is entirely correct. However the colloidal film, as I have used the term, is assumed to be wet and much thicker because of the absorbed water. The pressure, while causing many of the smaller particles to flow away, also causes the water in those parts of the film which receive the pressure to be squeezed out and hence reduces the capacity of this part of the film to shrink. Water is necessary to my explanation of the phenomena.

I might also add that I have found the same behavior in that part of a plastic German kaolin which remained suspended in water after standing 20 minutes.

THE PRODUCTION OF CHINA FROM DOMESTIC MATERIALS.

F. K. Pence: I am told that in the manufacture of Belleek china, the body is ground for two weeks in a drag mill. That amount of grinding is said to be essential to the successful manufacture of this very thin, translucent ware.

C. F. Binns: It is quite true that Belleek China must be ground very fine and the practice was at one time to do as Mr. Pence has stated. Of recent years the ball mill has been introduced, and the time of grinding has been much reduced. The fine grinding is essential both to the working of the clay and to the clarity of the body.

J. B. Shaw: There seems no doubt that this is the logical method of attacking the problem of determining the most fusible mixtures of the ingredients entering into a Bristol glaze as the results obtained by others working along the same line on other materials have shown.

The merits of this method of investigating glazes seem not to have been fully determined by this study. It is well known that the most fusible glaze is not necessarily the most desirable one, nevertheless the fusibility is always a major consideration, and no better method of studying this property has as yet been evolved.

It must be said in favor of this method that it simplifies the study of glazes in that they may be divided into three components, but it does not decrease the amount of work necessary to cover the field of Bristol glazes. However, in view of the fact that Rankin and Wright (*American Journal Science* XXXIX January, 1915) were able to predict the crystalline composition of Portland cement when working along this line, it would not seem at all impossible that the composition of the opacifying material in Bristol glaze could be determined by following out the same procedure. The necessary amount of work, however, would be enormous because of the number of ingredients entering the glaze.

THE COMPARISON BETWEEN SODIUM SULPHATE AND FREEZING TESTS FOR DRAIN TILE.

W. L. Howat: The strength of the solution as affected by a ten degree temperature change would probably be important if but one test were made, but in the test we conducted, the pieces were soaked in the solution for 24 hours and this was repeated five or six times, so that any slight tendency of the temperature to change the concentration of the solution would be taken care of by repeated tests. If there was one test on each piece, the effect would be more noticeable. The test is not expected to tear down a hard drain tile, that is not the idea. It will not distinguish between a hard drain tile and one slightly softer, but the idea is to be able to point out a drain tile that is too soft.

P. H. Bates: The author states — "the object of this part of the investigation was to determine whether a sodium sulphate test can be used for drain tile in place of freezing tests." Later he also says, "that the results for the concrete samples in test 'A' are so closely comparable to the freezing tests is probably due to the severe mechanical strain set up in this test by the crystallization of Na_2SO_4" — but then concludes "however sodium sulphate tests can not be considered a reliable substitute for freezing tests on concrete."

If he has shown that the sodium sulphate tests are as comparable to freezing tests in the case of concrete as in the case of clay tile, why cannot the former be considered a reliable substitute for the latter? There is no explanation given as to why the sulphate test is comparable to the freezing in the case of the clay test — but is it not due to "the mechanical strain set up in the test by the crystallization of Na_2SO_4"? Since in both cases the destruction of the specimen depends upon the force exerted by the growing crystals of either water (in the case of freezing) or sodium sulphate (in the case of the sulphate test), it is difficult to see why the sulphate test cannot be used advantageously as an "accelerated freezing test" for both classes of material.

It would appear as if the author is still under the influence of the work of Michaelis and Le Chatelier, according to which concrete in sulphate waters may be disintegrated by chemical reactions between the concrete and the sulphate. However, the reaction, while it may take place, has never been proven to be the cause of the destruction — but most evidence and the latest investigations show that the destruction is due — as the author states, "to the severe mechanical strain set up in this test by the crystallization." Framm[1] considers the same conditions beneficial that Le Chatelier and Michaelis consider harmful, as it renders the concrete more dense: Jeuett[2] has noted that the disintegration is due to the formation of crystals whose expansive force is enormous but which disintegrate to a fine powder on exposure to the air — Na_2SO_4. 10 H_2O(?). Klein and Phillips[3] state that the disintegrating action of calcium sulpho aluminate — the injurious compound formed according to Michaelis and Le Chatelier — has been greatly exaggerated: and Steik[4] could not find any compounds of hydrated calcium sulphate. In fact much more evidence can be cited, showing that the disintegration is mechanical than can be found showing it is chemical.

H. F. Staley: In reply to Mr. Howat, would say that a number of tests, as averaged, would give results for the average temperature, but unless care were taken to control the temperatures, the average temperature in the laboratory for one season of the year might vary by 5 or 10° centigrade, from the average temperature in the same or another laboratory at another season of the year. If this were the case, the results of sodium sulphate tests, made at the two different temperatures would not be at all comparable.

As Mr. Bates suggests, it might be feasible to neglect the possibility of chemical action in concrete tile, and to consider that the tile which are broken down have been destroyed by the mechanical strain set up by the crystallization of sodium

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[1] *Chem. Ztg.* 35, p. 256, 1911.
[2] *Proc. Am. Soc. Test. Mat.* 8, p. 480, 1909.
[3] *Tech. Paper No. 43*, Bur. of Standards.
[4] *Wyoming Sta. Reportt 1914*, p. 148-158; *Ex. Sta. Record*, 32, p. 787.

sulphate. However, there is always the possibility of chemical action between the sodium sulphate and lime compounds in the concrete. In the work described under method C of sodium sulphate tests, we have shown conclusively that some samples of concrete can be destroyed by simple immersion in sodium sulphate solution under conditions that preclude the possibility of the growth of sodium sulphate crystals. The amount of chemical action of sodium sulphate on a concrete sample will depend on the composition of the cement, on the density of the concrete, and especially on the extent to which the sample is protected by insoluble carbonates.

In this laboratory, at least, we are of the opinion that in any comparative testing work there should be no unknown variables. For this reason, I made the statement that sodium sulphate cannot be considered a reliable substitute for freezing tests on concrete.

THE RELATIVE ACTION OF ACIDS ON ENAMEL

J. B. Shaw: The author has, without doubt, eliminated one of the chief objections to his work in the last paper of the same title, i. e., the variation in the surface exposed. By correction of differences in specific gravity, the results obtained by this method would probably be sufficiently accurate to give an approximation of the relative solubility of "frits." However, even with this method there will be a very considerable variation in the surface exposed, and in dealing with frits of very low solubility it seems very likely that the percent variation of surface exposed would be sufficient to vitiate the results. For instance, if we have two frits whose relative solubility percent of area vary by only 10 percent it would only require a variation of 10 percent of area exposed to make the results of the experiment entirely wrong.

In view of the low solubility of frits it is obvious that the variation of the solubility will be very low and therefore the necessity for exact duplication of conditions for testing, in order to obtain even relative results, becomes very great. Therefore I am inclined to doubt the propriety of this method of obtaining the sample for testing. The unavoidable and immeasurable error introduced by this method of obtaining the sample could be avoided by casting a sample of the frit in a polished steel mold which would always give a plate of the same shape and size and consequently of the same surface. It would also seem that the action of acids on a surface of this nature would be much more nearly comparable to the action of the acid on the ware than the method used by the author. The cast plates could be annealed if necessary to prevent them from breaking, and in case they did break into two or three pieces, the area exposed could be readily calculated.

It should be said in passing also, that the title of this paper is somewhat of a misnomer since the frit is only the basis of the enamel except in the case of enamels that are applied to the ware in the powdered form, and even in this case the relative solubility of the frits can not be taken as the relative solubility

of the enamel after being fused onto the ware a second time. This fact was brought out by Mr. Landrum in the discussion of Part I of this paper. In the case of enamels which are applied wet and which invariably have considerable quantities of raw material added to the frit, it is quite obvious that this treatment will greatly modify the results when the enamels are treated with acids. Therefore we might suggest as a more appropriate title for this paper, "The Relative Action of Acids on Enamelled Frits."

H. F. Staley: It is a pleasure to note the persistence and care the author of this paper has shown in working up this test for determining the comparative solubility of enamel glasses in acids. It is true, as the author states, that the effect of differences of mill additions and burning might have a decided effect on the resistance of enamels when actually applied to the ware; but in developing an acid resisting enamel, the first requirement is to secure an enamel glass that is, in itself, resisting. For this sort of work the method of testing proposed by Mr. Poste seems to the writer to be entirely satisfactory. I have used the method in some work of my own and find it both rapid and accurate.

E. P. Poste: It has not been the claim of the author that the foregoing method is accurate for comparing the resistance of finished enamels to acids. Quoting from the first paper (Transactions Vol. 17, Page 138) "the object in devising a means of comparing the action of acid on enamel was to determine the relative action produced when an enamel is exposed to different strengths of the same acid, to comparing the various acids as to their relative action, to study the nature of the action of acids on enamel."

It would be very desirable to develop this method to a point where it would be of more practical application. As it stands however, we feel that it serves the original purpose by providing a means of studying the action of various solutions on a given enamel. Prof. Staley's remarks show further that it has possibilities as a means of comparing the resistance of various frits which as he has stated are the basis of the final enamels and must be right if the enamels are to be right.

KAOLIN REFINING

C. F. Binns: I take exception to the use of the word "blunging" when applied to the operation of ball mills. Mr. Sproat has used this expression in two entirely different connections. He speaks of blunging the clay with water in the process of refining and again of the "blunging" of a china mixture in which porcelain lined ball mills were used. This operation is grinding and not blunging and should be so designated. In this connection it may be remarked that Mr. Sproat's ball mills must be extremely inefficient: it is almost incredible that a grinding period of 20 hours should be called for. If the charge is proportioned to the size of the mill and the amount of water and the speed properly adjusted, there is no reason why successful grinding should not be done, even for china, in 4 hours or 5 at the most. Any practical man will condemn a necessity for 20 hours grinding because of the impossibility of keeping up the supply of material without an enormous equipment.

It remains to be seen whether the introduction of sulphuric acid for the reflocculation of the clay can be made without ill effects. Sulphuric acid is the worst enemy with which the whiteware potter has to contend and even a small amount remaining in the clay, whether free or as sodium sulphate, constitutes a serious source of danger.

The discussion which took place on the floor of the Society raised two points in this connection: one was the use of cobalt sulphate which is becoming very general. There is no doubt that this substance is subject to the same objections that have been mentioned. No direct evil has been traced to the use of cobalt sulphate simply because there are so many sources of sulphate that any single one can not be easily located.

The second question raised was as to the presence of sulphuric acid in glass in connection with the expulsion of this substance from a vitrified china. The behavior of glass is analogous to the behavior of glaze but not to that of the body. The evil of sulphuric acid in a clay is due to the fact that upon drying the whole of this impurity is concentrated upon the sur-

face. When burned to vitrification, the sulphuric acid can be expelled by the action of the softening silica. Glass being a molten mass affords no such facility though doubtless a considerable amount of sulphuric acid is expelled from the surface. That glass-makers are alive to the necessity of expelling the SO_3, is shown by the fact that when salt cake is used carbon in some form must be introduced to secure reduction and volatilization in the form of SO_2.

A. V. Bleininger: It might perhaps have been well to mention, in addition to the fundamental patent of Keppeler, the patent of Count Schwerin, who combines deflocculation of the clays with separation by electrical means. Mr. Sproat has worked out very ably the practical manipulation of the refining process. What he says agrees very well with the experience of Mr. G. H. Brown in installing such a process in Eastern Pennsylvania in 1914. (*Trans. Am. Ceram. Soc.* Vol. 17, p. 89). In the plant in question the alkali was added in the blunger and sedimentation took place largely in the troughs. No ceramist is employed in this instance as the separation took place sufficiently well within quite a range of alkali concentrations.

Some complaint has been made by potters that in jiggering a body containing clay refined in this manner some difficulty was experienced due to the "rubbery" nature of the clay. Whether this is true or due only to prejudice I am unable to say. In our own laboratory we have jiggered such bodies apparently without any difficulty.

H. Ries: Fluxing action in bricks during burning, is probably often less than many believe. I have often noticed that the sections of vitrified brick, which have been burned the normal time, show angular particles, which show little or no corrosion due to fluxing.

Mr. Teeter referred to the presence of mica in one slide. The fluxing action of mica depends of course upon its fineness. Mr. Stull, some years ago, in a paper before the Ceramic Society, spoke of the fluxing action of mica at a comparatively low cone. If, however, the individual scales are sufficiently large to be noticeable, they may retain their individuality up to cone 10 or 12, as I pointed out in my report on the New Jersey Clays.

I would also like to ask Mr. Teeter if he was able to identify any other minerals in the finer grained portions of the burned clay.

Mr. Teeter replied that at the highest temperature, one of the clays seemed to be a little glassy, and showed an agglomeration of very fine needles which might be sillimanite.

H. F. Staley: The main point for discussion raised by this paper seems to be whether the viscosity test is of service in determining the value of clays for the manufacture of vitrified products. There seems to be a tendency to multiply the number of laboratory tests on clays and clay products. When once these tests come into vogue, it is necessary for testing laboratories to apply them whether they have much real value or not.

In his final conclusion, the author is quite conservative, stating that the viscosity temperature curve is capable of giving additional data in differentiating between shales for the manfacture of vitrified products, such as paving brick, and those not suited for such products. In the body of the paper he says —"the curves of the shales which are suited for the manufacture of vitrified brick do not show any abrupt break, or decrease in the viscosity, below the temperature of Seger cone 5. The curves of the shales not suited for vitrified ware show a break in the viscosity curve or a decrease in the viscosity at some tem-

perature below Seger cone 5." He does not state which clays he consders suitable for the manufacture of vitrified products, but Nos. 234, 235, and 239 are used in the manufacture of paving brick and No. 231 is, in the opinion of the author, based on results of the curves shown in this paper, suitable for the manufacture of vitrified brick.

It has been usual among ceramists to assume that those clays whose porosity decreases at a gradual rate to a small amount at commercially feasible temperatures are, as far as burning properties go, worthy of testing on a large scale for the manufacture of vitrified products. According to this criterion also the above four shales would be classed as safely vitrifying clays, although there would be some question about No. 234, due to the sudden drop in porosity between cones 3 and 5.

According to the rule stated by the author, clays Nos. 232, 233, and 238 would also be suitable for the manufacture of vitrified products. The sudden drop in porosity of clay No. 232 between cones 3 and 5, and of No. 233 between cones 5 and 7 would raise a doubt in the mind of any ceramist as to the availability of either of these clays for use alone in the manufacture of vitrified products. The immense drop in the porosity in clay No. 238 between cones 06 and 03 would preclude its use for vitrified products. In these three cases then, the old and well tried porosity determination seems to be a better indication of the usability of the clay, as checked by the purposes to which the clays have been put, than the viscosity test.

Clays Nos. 236 and 237 are shown to be of poor quality by both the porosity figures and the figures for the viscosity determination. These figures are in accord with the uses to which these two clays are actually put. Clay No. 230 looks fairly good according to the porosity figures but is condemned by the viscosity curve. Since this clay is used for the manufacture of common brick only, we may assume that it is not suitable for the manufacture of vitrified products, and that in this case the viscosity curve indicates the poor quality of a clay when the porosity does not.

Summing up then, we may say that the porosity and viscosity check each other in six cases, that the viscosity curves are

wrong and the porosity curves right in three cases, and the viscosity curve right and the porosity curve wrong in one case. In as much as it has been amply demonstrated by experience that the only way to really determine whether a clay will make vitrified products, such as paving brick, is to make actual full size clay products of the kind to be manufactured, it is a question whether it is desirable to add the viscosity determination to the list of preliminary tests for clays.

Of course the so-called viscosity determination is not really a new test, it is simply another way of conducting the old warpage test, in which an entire bar of clay was supported on each end by a piece of fire clay and sagged down in the middle when it commenced to soften. From the standpoint of the mechanics, they are really both tensile strength tests and the two methods are used as alternatives in determining tensile strength. The formula for the method corresponding to the warpage test is:

$$S = \frac{P}{a}$$

S = tensile strength in pounds per sq. inch.
P = load in pounds.
a = area of cross section in sq. inches.

The formula for the test corresponding to the so-called viscosity test is:

$$S = \frac{Mc}{I}$$

S = tensile strength in pounds per sq. inch.
M = bending movement in inch pounds.
c = half height of beam in inches.
I = moment of inertia of cross section.

The so-called viscosity test is really a warpage test which is accelerated by the fact that a heavy load is suspended from the piece. As far as the writer knows, there has been published

no data showing the actual correlation between the viscosity determinations and the tendency of clay wares to warp.

P. Teetor: In reply to Dr. Ries' statement I am pleased to say that since this manuscript was sent to the editor, Dr. W. P. Haynes, professor of mineralogy and petrography, who has just recently joined the geology faculty at the University of Kansas has examined the thin sections prepared from the burned specimens of these shales and reports as follows:

Thin sections were made from the test samples after cooling in the kiln. For samples Nos. 230 to 238 inclusive, three sections were made as follows: section 1 from cone 06 temp. 990°C.; section 7, cone 3, temp. 1110°C., and section 13, cone 8, temp. 1160°C. Six sections were made of No. 239 taken at intermediate stages from cones 06 to cone 8.

The color changes in these sections are very interesting and rather instructive. Section 1 is in every case of a bright brick red color, due to the complete oxidization of the iron to the ferric state. In a few cases such as section 1 of No. 239 there is more or less of a yellowish limonite discoloration on some of the minerals which partly masks the red color. Section 7 is frequently mottled red and brown in color, or else a deep hematite red. The mottled appearance is evidently due to the partial alteration of the iron from the ferric to the ferrous stage. This partial change is also shown in section 10 of No. 239, from cone 5, at a temperature of 1125°C. Section 12 of the same sample from cone 7, temp. 1140°C. is completely changed to a dark brown color as are all of the sections numbered 13. These therefore clearly show that the change of the iron from the ferric to the burnt ferrous state takes place between 1100° and 1130° in these clays.

The sections indicate a wide variation in the composition of the clays, especially in the quartz content. Measurements were made on the grains of quartz both as to size and as to relative abundance. The results are as follows:

r = rare.
c = common.
C = very common.

No.		
230	.0177mm av.	r
231	.0236mm av.	r
232	.0354mm av.	r
233	.0431mm av.	c
234	.0431mm av.	C
235	.0472mm av.	C
236	.0431mm av.	c
237	.0431mm av.	c
238	.0118mm av.	r
239	.0236mm av.	C

Irregular shapes. Wide range in size up to 1mm.

There is a gradual increase in the quantity of glass with all of the series of sections. Little glass and much opaque groundmass, chiefly iron-stained kaolin, is present in section 1. Quartz is the most abundant mineral, but a few of the samples contain some mica, notably Nos. 232, 234 and 239. Feldspar is not very abundant in any of the samples and is seldom recognizable after section 1. In section 7 there is much more glass and therefore greater transparency to the section. Some of the glass contains small bubbles and it frequently is seen to surround open spaces which were present in stage 1. Section No. 13 shows the most interesting changes. There is in most of the samples a large amount of glass in the groundmass. This glass is more or less filled with quartz grains in various stages of fusion. Usually they are only affected on the border, but in a few cases the outline is very irregular and the glass appears to run deeply into the grains. The mica is not recognized after No. 7 stage, and is probably fused before 13. In places there appear cloudy spots in the glass which show very minute crystal aggregates which could not be identified.

The amount of glass in all of these samples is large at cone 8. Samples Nos. 230, 231 and 238 appeared to be less vitreous than the others. Owing to lack of time in studying these sections the writer is obliged to stick to general observations, but he is convinced of the great possibilities of microscopic study in getting at the character of the changes in clays at different temperatures,

and hopes to have the opportunity to carry on this study much further with the assistance of Mr. Teetor.

P. Teetor: Mr. Staley has raised the point as to whether the viscosity test is of service in determining the value of clays for the manufacture of vitrified products, and according to his analysis finds that it is not as valuable a test as the porosity temperature test, and finally questions the desirability of adding this test to the list of preliminary tests for clays.

He goes on to say that "those clays whose porosity decrease at a gradual rate to a small amount, at commercially feasible temperatures are, as far as burning properties go, worthy of testing on a large scale for the manufacture of vitrified products." He gives no limits for the terms "gradual rate" and "small amount." Mr. Purdy's limits, as given in Bulletin 9 of the Illinois Geological Survey, are the most accurate I have seen published, but any one who has investigated a large number of clays undoubtedly has found high grade paving-brick clays with porosity temperature relations falling outside these limits.

If the same leniency is allowed the viscosity curves of these ten shales as is necessary to allow the porosity temperature curves, in order that they may check the facts, it will be found that viscosity curves are at least as accurate, if not more accurate, than the porosity temperature curves. In fact, it is practically safe to say that the viscosity curves of shales suited for the manufacture of vitrified brick do not show any abrupt break or decrease in the viscosity below a temperature of Seger cone 3. By this criterion clays Nos. 231, 232, 233, 234, 235, and 239 are suited for the manufacture of vitrified products.

Mr. Staley's statement that, "according to the viscosity curves shale 238 would be suited for the manufacture of vitrified products," is erroneous, because the curve shows a very distinct decrease in viscosity at a temperature of cone 03. His fears for the sudden drop in porosity of shale 232 between cone 3 and 5, and shale 233 between cone 5 and 7, are not well founded, because there are quite a few first-class paving-brick shales which show as large a drop in porosity between these cones. Mr. Purdy's work on the Illinois clays in Bulletin 9 of the Illinois Geological Survey, also shows this to be true. As a matter of

fact I have made paving block of shale No. 233 with a small laboratory auger machine and burned them in a laboratory test kiln in three days' time, and the rattler loss in the standard rattler was 18.78 percent.

Analyzing the viscosity curves with cone 5 as the critical temperature for the decrease in viscosity, we find shales Nos. 232, 233, 234, and 239 are suited for the manufacture of vitrified products. With cone 3 as the critical temperature we find clays Nos. 231, 232, 233, 234, 235, and 239 suited for the manufacture of vitrified products. Examining the porosity temperature curves and comparing them to the limits laid down by Mr. Purdy, as referred to above, we find that at cone 06 shale 232 is the only one lying within these limits, at cone 03,—Nos. 232, 236, and 239, at cone 1,—Nos. 231, 232, 233, 234, and 239, at cone 3,—Nos. 231, 232, 233, 234, 235 and 239, at cone 5,—Nos. 230, 234, 235, and 238, and at cone 7,—No. 238. It is known that shales Nos. 233, 234, 235, and 239, are good paving brick shales. In as much as No. 232 is located immediately beneath No. 233 and is used as a portion of the mixture represented by No. 234, we are reasonably safe in assuming that paving brick can be made from No. 232. Shale No. 231 has never been used for anything except hollow building tile, but both porosity and viscosity curves indicate that there is a possibility of its being used for the manufacture of vitrified products. Therefore, from the above comparison it is evident that the viscosity curves of these shales give data which is as valuable as the porosity temperature data, and is desirable as an additional check in determining the value of such shales for the manufacture of vitrified products.

The principal objection to the viscosity test, as developed by preliminary work done to date, is its time consuming nature. There is no doubt as to the value of the viscosity data, and it is to be hoped that some shorter method can be devised for securing data which will be as accurate as is secured by the present method.

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